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Journal of the American Ceramic Society

Volume 2

Containing the papers read at the meeting held at Pittsburgh, Pa., February 3rd, 4th, 5th and 6th, 1919, together with supplementary discussions and other matters

George H. Brown, Editor

This Society is not, as an organization, responsible for the statements of facts or opinions expressed in these papers and discussions

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JOURNAL
OF THE
AMERICAN CERAMIC SOCIETY

A monthly journal devoted to the arts and sciences related to the silicate industries.

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EDITORIALS.

COÖPERATIVE RESEARCH.

The comprehensive plan for the organization of a refractories research association, as outlined in this number of the JOURNAL by Dr. Washburn, merits careful consideration and thorough discussion. The fact that similar research organizations have been fostered by groups of manufacturers in other industries and have been found worth-while, both in the advancement of technique and from the standpoint of a financial investment, is a strong argument for associations of this kind.

It would appear that the ceramic industry, comprised as it is for the most part of small units, offers a promising field for research associations, not only as related to refractories, but in the several branches of the industry. The stimulated interest on the part of our manufacturers in technical advancement is evidenced by the increased support which they are lending to the American Ceramic Society and related organizations and by the unusual demand for men with technical ceramic training. The old secretiveness and rule of thumb methods are gradually giving place to coöperation and exchange of ideas and experiences. The support which the Refractories Manufacturers' Association is giving to the maintenance of a research laboratory of its own is a step in the right direction. An avenue for the further advancement of this and other branches of the industry will undoubtedly be found through the recently organized Professional Divisions of the Society.

Some of the difficulties to be encountered in launching organizations of this kind may appear insuperable at the start but a co-

ordination of the activities of our present research laboratories would be a long step toward the consummation of the greater and ideal research organizations.

LOCAL SECTIONS.

The following telegram, recently received by the Chairman of the Membership Committee, is indicative of the interest taken in the Society by workers in the silicate industry in all parts of our country:

Los Angeles, Calif., February 12, 1919.

There are about a dozen good men in town and more in the vicinity who should become members of the American Ceramic Society. Please send descriptive literature, application blanks and full information at once. There is a possibility also of a Western Section in which affiliated lines would coöperate. Send complete data. P. A. B.

The Chairman reports the same interest shown from Toronto, Canada. Such interest on the part of individuals bespeaks the organization of more Local Sections, stronger Professional Divisions and a larger membership than even the most optimistic of us have visioned.

CERAMIC ENGINEERS.

Although with the curtailment of war activities, there appears to be no dearth of chemists and engineers in most lines, the demand for men with technical ceramic training appears to be as acute as during the last year of the war. This is significant, in view of the present period of reconstruction and consequent marking time in the industry. The comparatively small number of men to be graduated from the ceramic departments of our colleges and universities at the close of the present school year will not fill the vacancies, and the demand will be felt for some time to come.

In order that an adequate and continuous flow of technically trained men be available for the future requirements of the industry, the enrollment in our ceramic schools should be increased. A normal increase is to be expected but it is to be hoped that some means will be found to enlist the interest of a greater number of prospective college students in the possibilities for advancement in the silicate industries.

ORIGINAL PAPERS AND DISCUSSIONS.

REFRACTORY MATERIALS AS A FIELD FOR RESEARCH.

A Survey of the Scientific Aspects of the Subject.[1]

By Edward W. Washburn, Chairman, Committee on Ceramic Chemistry, National Research Council.

1. **Definition of the Term Refractory Material.**—For the purpose of defining an homogenous class of materials for systematic investigation, the term "refractory material" *will be here understood* to signify any non-metallic material capable of withstanding elevated temperatures, without destruction or deterioration (by fusion, sublimation, chemical decomposition or physico-chemical transformations) so rapidly as to preclude its use in the construction of vessels, linings, furnace walls, flues, etc., subjected to high temperatures. Although resistance to high temperatures is the primary and distinguishing characteristic demanded of refractory materials as a class, almost every refractory employed in modern industry must also exhibit, while at a high temperature, an adequate resistance toward one or more of the following destructive agents: (1) pressure or load; (2) mechanical vibration; (3) frequent, rapid and unequal heating or cooling, or any one or more of these; (4) the stresses set up by expansion or contraction of other parts of the furnace or vessel of which the refractory material is a component part; (5) mechanical abrasion by ashes, cinders, etc., or by the furnace charge itself; (6) the chemical action of atmospheric and furnace gases; (7) the slagging action of the furnace charge or of materials given off by it; and (8) the

[1] This report was drafted under the auspices of the Section of Industrial Research of the National Research Council, as a preliminary step in the consideration of the nature of the problems involved in research in this field and of the possibilities of attacking them by concerted action.

chemical action of any other furnace parts such, for example, as the electrodes or the heating element in an electric furnace; and in certain special cases the refractory must (9) while at high temperature (*a*) remain a good electrical insulator, or (*b*) become an electrical conductor; or (10) (*a*) remain a good thermal insulator, or (*b*) become to a given degree a thermal conductor.

2. **The Importance of the Subject.**[1]—Every industrial plant which employs high temperatures in any part of its work has a more or less acute problem of refractory materials to deal with. The refractory materials employed may vary all the way from the ordinary fire bricks (such as are employed in the boiler settings of the power plant) to highly specialized materials designed to withstand one or more of the special destructive agents mentioned above. Railway locomotives and the power plants of ships can be operated with the highest efficiency only when properly designed refractory materials are used in their construction.

Practically all of the metallurgical industries, both those which have to do with the extraction of metals from ores as well as those engaged in working the various metals or preparing alloys, have especially trying and difficult refractory problems to meet. An important part is also played by refractory materials in the manufacture of electric furnace products (such as abrasive materials, graphite, carbide, nitrogen products from the air, and a variety of other chemical products); of glass and quartz articles; of lime, cement, potash, fuel gas, ammonia, coke, and many of the pigments; and of course of all ceramic products.

As a field for industrial research, the subject of refractory materials is, therefore, fundamental in character and widespread in its practical applications. The present trend of many industries in the direction of using increasingly high temperatures[2] in their operations is a further indication of the growing importance of

[1] See also Geo. A. Balz, "Why Refractories Are a World Necessity," *Brick and Clay Record*, 48, 741 (1916).

[2] "It will be recognized by all who have studied the matter closely that the future industrial success of any country will largely depend upon the extent to which it develops high temperature processes." Cantor Lecture, by C. R. Darling, before the Royal Society of Arts, London, February, 1918.

a thorough and systematic scientific investigation of the various problems connected with the preparation and use of refractory materials.

3. **Conservation of Fuel.**—In a high temperature furnace or kiln, where the internal temperature required is higher than the refractory lining will withstand, it is customary to protect this lining by artificial cooling either by means of air or water. Such artificial cooling naturally results in a great waste of heat and in a corresponding greater consumption of fuel. The ideal arrangement would be to cover the outside of the furnace with a good thermal insulator so as to retain this heat in the furnace, but in many cases under present conditions such an insulation would result in the rapid destruction of the refractory lining, owing to the fact that this lining would soon attain the temperature of the inside of the furnace. It is obvious that the development of refractory materials which would permit the thermal insulation of industrial furnaces would result in an enormous fuel saving since the wastage resulting from present methods is one of the large elements in the total fuel consumption of industrial furnaces. Perfect adaptation of the refractory to furnace conditions (temperature, pressure, chemical action, mechanical abrasion, etc.) is one of the big problems whose successful solution would constitute a great contribution to the fuel conservation movement.

4. **Annual Production and Consumption of Refractory Materials in the United States.**—Complete statistics on the annual production of refractory materials and products in the United States do not seem to be available in public records but from the data at hand the value of the annual production of such materials may be safely estimated as greater than (60) sixty million dollars.

The following statistical data on the subject have been recently compiled at the writer's request from the records of the United States Geological Survey:

Bauxite refractories.—In 1917 the bauxite used for the manufacture of refractory wares was about 12,000 tons. Approximately three (3) million bricks $2^1/_2 \times 4^1/_2 \times 9$ inches were sold, valued from $50 to $380 per thousand. In 1917 the bauxite used in the manufacture of refractory wares was over 2,500 tons,

but exact figures are not available, nor is information as to the quantity or value of the products made.

Refractories manufactured from quartz, chert, silica, fused silica, etc.—Except as elsewhere noted concerning silica brick, the Geological Survey has no record of the manufacture or production statistics of refractory wares made of these materials. Only the total output of silica in various raw forms is known, and this is not separated or classified according to uses.

Refractories manufactured from ganister.—There is no available record of the refractory wares and materials made of ganister. The Survey's record of ganister production—that is, sales of quarry products—is as follows:

PRODUCTION 1913–1917.

	Short tons.	Value.
1913	.....	$ 376,775
1914	.....	288,244
1915	573,304	336,267
1916	859,956	529,805
1917	1,001,630	1,117,558

Mica schist for furnace linings.—The following data concerning the production of mica schist for furnace linings are available:

PRODUCTION 1913–1917.

	Short tons.	Value.
1913	....	$45,102
1914	....	54,567
1915	....	24,625
1916	33,236	47,304
1917	39,975	85,986

Magnesite refractories.—Statistics of the quantity and value of magnesite produced and sold in the United States are available, but no information is at hand as to the proportions of this material which enter into refractories, magnesium chloride, magnesia, alba, manufacture of paper and other products. The Geological Survey assumes that the only source of information as to the quantity of refractory brick and shapes made from magnesite in the United States would be by a canvass of the manufacturers.

Dolomite refractories.—The Geological Survey has only the following statistical information:

ESTIMATE OF DOLOMITE AND EQUIVALENT IN "DEAD BURNED" LIME, PRODUCED FOR REFRACTORY PURPOSES 1914–1917.

	Dead burned. Short tons.	Unburned. Short tons.
1914	13,053	26,058
1915	50,223	100,446
1916	136,240	267,446
1917	176,876	234,720

Chromite refractories.—There is no available information on the quantity and value of chromite used in the manufacture of refractory wares, or on the value of the refractories manufactured, other than statistical data showing that 4364 long tons of chromite in the form of refractories were consumed during the first half of 1918 and that 1959 long tons were similarly consumed during July, 1918.

Graphite refractories.—So far as known to the U. S. Geological Survey, there are no statistics showing quantity of molded graphite articles, such as graphite crucibles, anywhere available for the years asked for, or for that matter, for any periods.

Quartz-glass and fused silica refractories.—No available data.

Zirconia refractories.—Data concerning the total annual production of zirconium minerals are contained in the Annual Mineral Resources Reports.

Rare earth refractories.—The following information is taken from the report on the gas-mantle industry now in the files of the United States Tariff Commission:

PRODUCTION AND IMPORTATION OF THORIUM NITRATE IN THE UNITED STATES, 1913–1917.

Year.	Quantity produced. (Pounds.)	Quantity imported. (Pounds.)
1913	Information	112,105
1914	confidential	144,413
1915	cannot	78,516
1916	be pub-	22,261
1917	lished.	1,877

Clay Refractories.—The following statistics on the value of clay refractories manufactured in the United States for the years 1913 to 1916 have been compiled by the United States Geological Survey:

Product.	1913.	1914.	1915.	1916.
Fire brick, including refractory block or tile, boiler and locomotive tile, tank blocks and similar refractory products..	$16,811,316	$13,476,022	$15,800,062	$25,155,519
Other fire brick, including some special shapes, etc.....	134,635	115,144	121,747	311,052
Silica brick, including clay-bond and lime-bond brick.....	3,815,806	2,951,525	3,039,869	5,650,610
Zinc retorts.....	(*c*)	576,655	823,545	1,553,691
Zinc condensers.....	(*d*)	176,591	260,436	512,453
Glass melting pots and other glass-house refractories (Special effort to collect statistics of these products from the consumer manufacturing for his own use was not made prior to 1915.....	568,603	498,096	719,889	1,989,754
Gas retorts.....	65,846	41,372	23,835	35,821
Charcoal furnaces (portable).....	37,217	36,243	32,865	27,280
Muffles, scorifiers, assay supplies and crucibles (other crucibles are included with chemical porcelain and chemical stoneware).....	63,869	67,367	98,105	364,563

Saggers (prior to 1917 statistics for saggers were not collected from the sagger consumer manufacturing for his own use)	(e)	(e)	(e)	34,476
Chemical porcelain and chemical stoneware	(f) 224,894	(f) 246,918	(f) 620,401	1,054,061
Potters' supplies (pins, stilts, and spurs)	125,987	130,740	126,780	188,643
Mantle rings and special ware for gas lighting and heating, including magnesia ware and refractory porcelain for electric ranges and heaters (including a small valuation for pins and stilts)	(e)	(e)	172,261	220,849
Undistributed refractory products	364,519	329,423	22,288	.....
Total	$22,212,692	$18,646,096	$21,862,083	$37,098,772

(c) Reported by one producer only for 1913, and included with "Undistributed refractory products"—statistics for zinc retorts were not collected prior to 1914.

(d) Reported by one producer only for 1913 and included under Miscellaneous—statistics for zinc condensers were not collected prior to 1914.

(e) Reported by less than three producers—included in "Undistributed refractory products."

(f) Chemical porcelain and chemical stoneware, not separately classified prior to 1916, were probably partly reported under Stoneware and *Yellow* and Rockingham ware in 1913, 1914, and 1915.

Free Public Library, Newark, N. J.

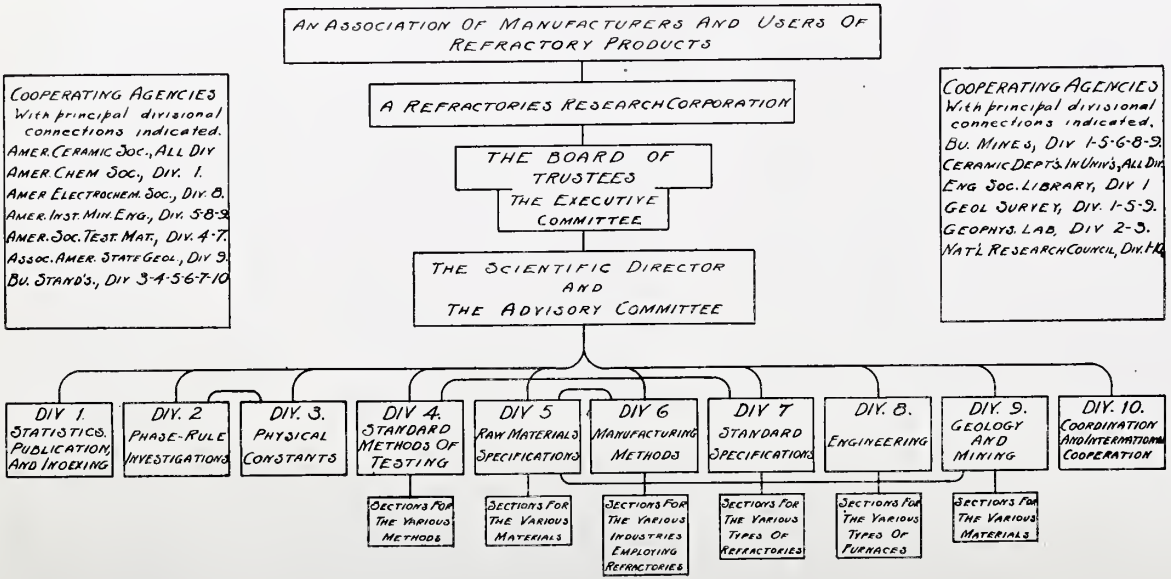
5. An Organization for the Prosecution of Research in Refractory Materials.—In outlining and describing the various aspects of the subject of refractory materials as a field for research, we shall first create, for convenience of presentation, a hypothetical organization, which we shall assume proposes to engage in a complete and systematic study of *all problems* connected with the nature, preparation, properties, and industrial application of refractory materials. It will be understood, therefore, that the particular organization described below is a purely fictitious one created on paper, merely as a convenient machine for setting forth in systematic fashion the various scientific aspects presented by the subject of refractory materials as a field of research. It is not suggested that this organization, created here as a convenient means of classifying the different aspects of our problem, would be the most suitable type of organization for actually undertaking to carry out research in this field, since in constructing an actual working organization various practical questions would have to be considered which do not enter into a plan of organization designed merely to display the different scientific aspects of the problem. It is not the writer's intention to propose at this time any particular type of working organization since the formation of such an organization would require the combined labors of a group of experts familiar with all of the practical questions involved. It is hoped, however, that the type of organization employed here, for the purpose indicated, will, in so far as it sets forth in a systematic manner all of the various scientific aspects of the subject, be convenient as a starting point in planning some practical working organization for undertaking the solution of the many important questions which our subject presents.

With this explanation therefore we shall assume that our hypothetical research association or corporation finds it convenient to organize itself into divisions for handling the different parts of its work. These divisions might be somewhat as follows:

A. The Board of Trustees and the Executive Committee.
   (1) The Division of Statistics, Publication and Indexing.
   (2) The Division of Phase-Rule Investigations.
   (3) The Division of Physical, Chemical, and Ceramic Properties of Raw Materials and Manufactured Products.

ORGANIZATION OF A REFRACTORIES RESEARCH ASSOCIATION

Designed to display the various subdivisions of the field and their interrelations.
Not proposed as the best immediate form for a working organization.



(4) The Division of Standard Methods for Testing Refractory Products.
(5) The Division of Raw Materials Specifications.
(6) The Division of Manufacturing Methods.
(7) The Division of Standard Specifications for Refractory Products.
(8) The Engineering Division.
(9) The Division of Geology and Mining.
(10) The Division of Coördination and International Cooperation.

6. The Board of Trustees and the Executive Committee.—The Board of Trustees would be charged with the raising and expenditure of all funds and would exercise general supervision over the financial affairs of the Association. It would elect the Scientific Director and on his nomination appoint the chiefs of all Divisions.

The Executive Committee of the Board would be empowered to act for the Board in the intervals between Board meetings. After receiving the recommendations of the Advisory Committee, it would prepare the annual budget for presentation to the Board of Trustees and would also prepare the program of business for each meeting of the Board.

7. The Scientific Director and the Advisory Committee.—The Scientific Director would exercise general supervision over all of the research work of the Association. He would make all nominations for heads of Divisions and would transmit to the Executive Committee, with his recommendations, all nominations for appointment to positions on the research staff. For a time at least, he might also act as the head of one of the Divisions since he would presumably be specially qualified as an expert in at least one of the principal lines of work of the Association.

The chiefs of all Divisions, together with possibly one or two additional members of national reputation as experts in industrial research, would constitute an Advisory Committee to the Scientific Director. This Committee would be charged with the preparation of the annual program of research and the necessary budget

for accomplishing it. This program and budget would be transmitted by the Director to the Executive Committee of the Board of Trustees, which body would exercise the final judgment concerning the program and budget.

8. Division 1. Statistics, Publication and Indexing.—The duties of this Division would be as follows:

(1) The preparation of an exhaustive classified bibliography of the extensive and widely scattered literature on the subject of refractory materials and all matters relating thereto. This bibliography should be indexed and cross-indexed to the fullest degree so that the literature might be utilized to the best possible advantage. After publication this bibliography should be kept up to date by means of annual or semi-annual supplements which might be combined decennially into new editions.

(2) By coöperation with the American Chemical Society, the work of securing and publishing abstracts of all papers dealing with any aspect of the subject of refractory materials should be promoted and improvements secured in the abstracting and in the arrangement and cross-indexing of the abstracts in the pages of *Chemical Abstracts*.

(3) To supplement the above bibliographic material, the Division might arrange with competent experts for the preparation and publication, from time to time, of critical digests in the form of monographs on selected topics. Each of these monographs should be an exhaustive, critical presentation and discussion of all the essential known facts concerning the subject matter and should contain a complete set of tables of numerical data and a complete bibliography. In this way, it would be possible to build up gradually a library of authoritative and trustworthy works of reference dealing with all the various aspects of refractory materials and their uses.

Each important refractory material (silica, magnesite, kaolin, alumina, etc.) might become the subject of such a monograph as might also the practice and requirements of each type of industry employing refractories. Not the least value of such a set of monographs would be its revelation of the relatively small amount of really reliable scientific data available on the subject

of certain refractories and the enormous amount of work which still remains to be accomplished. The preparation of such a monograph should precede the undertaking, on any extensive scale, of a program of research on any given topic.

(4) The biennial publication of a handbook of tables of physical, chemical and ceramic constants relating to refractory materials and products as well as standard methods for testing and examination would be valuable. In the preparation of such a handbook, this Division would work in coöperation with Division 3.

(5) Either through the medium of a journal of its own, or preferably through arrangements with one or more established journals, the Division should provide for the publication of all investigations carried out under the auspices of the Association, in all cases where such publication has been approved by the Board of Trustees.

(6) By coöperation with the appropriate governmental agencies, such as the Bureau of the Census and the U. S. Geological Survey, the Division should endeavor to secure improvements in the collection of statistics relating to refractory raw materials and manufactured products, to the end that all valuable statistical information on this subject should be properly collected and classified.

(7) This Division might also become responsible for the more general diffusion of accurate information concerning the manufacture and use of refractory materials and to this end might inaugurate a publicity program for educational purposes.

9. **Division 2. Phase-Rule Investigations.**—This Division would have general charge of the initiation, promotion, and direction of the most fundamental as well as the most difficult and expensive scientific studies which are required in building up our scientific knowledge of the refractory materials themselves.

As regards the common characteristic possessed by all refractory materials, that of resistance to high temperature, the initial problem presented for experimental investigation is to a large degree a problem in physical chemistry involving as its most important feature the application of the Phase-Rule and the laws of solutions. Thus, in accordance with the known laws of

physical chemistry, the effect of the presence of impurities in a refractory material is always to decrease the refractory power,[1] except when the proportion of the impurity is so large as to cause the composition of the mixture to coincide with a maximum point in the phase-rule diagram for the system, under which conditions the effect of the "impurity" may be either a decrease, an increase, or no change at all in the refractory power, depending upon the materials involved. According to the same laws, it is also in nearly all cases true that the larger the number of materials employed in the manufacture of a refractory product, the lower will be its refractory power. A great many of the patents issued for refractory materials cover products whose manufacture violates all of the principles mentioned above. These principles are also violated in the rather widespread idea that, as a general rule, the refractory power of a given material can be increased by mixing with it a second, *more refractory material.* Indeed the various formulas, which are given in text books and treatises on ceramic subjects, for calculating the refractory power (*i. e.*, softening point) of a mixture from its composition and the melting points of its components, seem to have been formulated without regard to established physico-chemical laws.

An illustrative example.—In order to illustrate the importance and the fundamental character of the physico-chemical relations displayed by the phase-rule diagram, in the solution of problems connected with the manufacture and use of refractories, a brief consideration of such a diagram for the system, magnesia-alumina, will be given here. In other words, given the two substances magnesia, MgO, and alumina, Al_2O_3, what are the possibilities, as displayed by the phase-rule diagram, of manufacturing refractory products from these two materials? The phase-rule diagram for this system is shown in Figure 1.[2] In this figure temperatures are indicated vertically and compositions, expressed in weight per cent, horizontally.

Both magnesia and alumina are separately employed in the industries as refractory materials. Alumina, however, at present

[1] For a statement of the factors which determine the fusibility of a material, see Washburn, *Trans. Am. Ceram. Soc.*, **19**, 195 (1917).

[2] See Rankin and Merwin, *J. Am. Chem. Soc.*, **38**, 571 (1916).

market prices is considerably cheaper per pound than magnesia. Either substance when employed alone presents the difficulty of finding a suitable bonding material to use when shaping into the desired form, since any foreign bonding material which remains in the product after firing results in a lowering of the refractory power, while a bonding material which burns out during

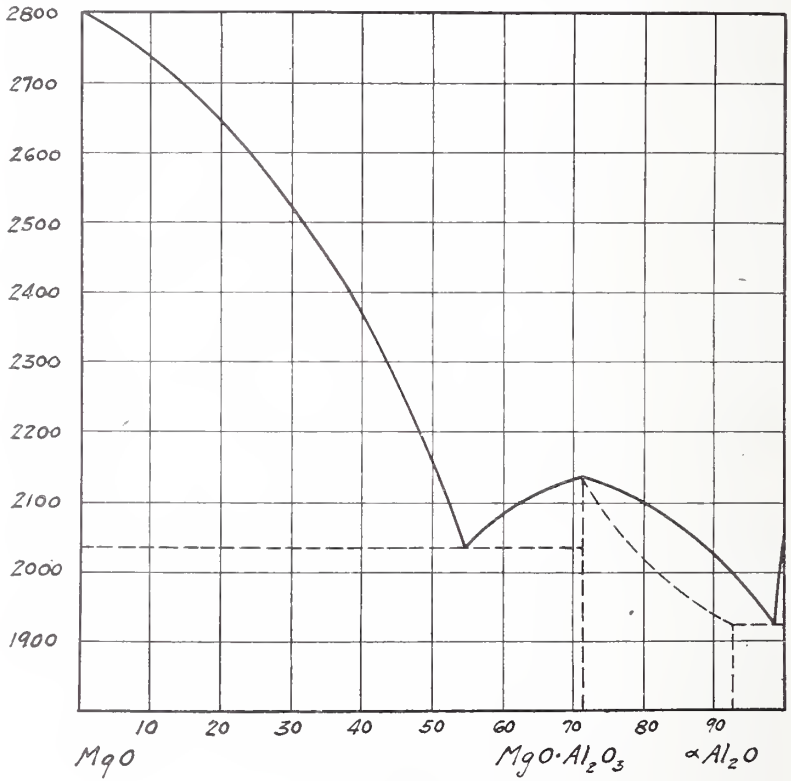


FIG. 1.

the firing leaves a loose structure with insufficient mechanical strength for many purposes.

The diagram shows that pure magnesia will not liquefy until a temperature of 2800° C is attained. In other words, as far as its mere resistance toward softening at high temperatures is concerned, magnesia is one of the most refractory substances which we

have. Although no liquefaction occurs below 2800° C, considerable deterioration occurs at much lower temperatures owing to crumbling, spalling, and sublimation, the volatility of the magnesia being so high that at high temperatures the magnesia vapors from a furnace lining will fill the whole interior of the furnace and condense upon everything contained therein. This characteristic makes it undesirable to use magnesia as a refractory for many purposes.

In order to increase the mechanical strength of a refractory body, both in the green state and after firing, it is frequently customary to mix with the principal constituent a small amount of a second constituent to act as a bond. For example, let us suppose that small amounts of alumina were mixed with magnesia for this purpose. The problem then presents itself as to the magnitude of the effect of such admixtures upon the refractory power of the magnesia. The answer to this question can be obtained from the phase-rule diagram. The curve in Fig. 1 shows that if any quantity of alumina, no matter how small and not exceeding 70 per cent, be mixed with the magnesia, the liquefaction temperature, that is the temperature at which liquefaction will *begin*, drops at once from 2800° to 2030°. In other words, alumina, present as an impurity or added for bonding purposes to magnesia, always decreases the initial liquefaction temperature by 770°.

This initial liquefaction temperature is known as the "eutectic temperature," and the first portions of liquid which appear when this temperature is reached will not have the composition of the original body, but instead will have what is known as the eutectic composition, that is, the composition corresponding to the eutectic point, which for the case under consideration is shown by the diagram to be 55 per cent of alumina and 45 per cent of magnesia. The maximum amount of this liquid which can be formed by heating the refractory body to any given temperature can also be readily calculated from the diagram. The resultant weakening of the mechanical strength of the refractory body depends of course upon the amount of this liquid which is formed. A small quantity of liquid might be held in the pores of the body without decreasing very greatly its mechanical strength, but as the amount

of the liquid increases the strength of the body continually decreases until finally a cone made of the material is no longer able to stand up under its own weight, owing to the fluidity produced by the presence of the eutectic liquid in its pores.

Turning now to alumina, we notice from the diagram that a refractory body made of pure alumina will show no liquefaction whatever until a temperature of 2050° is reached, while the admixture of any quantity of magnesia not exceeding 30 per cent by weight with the alumina will give a body which begins to liquefy at 1925°, which is the eutectic point on the right of the diagram. It is evident that alumina as an impurity in magnesia produces a much greater lowering in the initial liquefaction temperature than does magnesia as an impurity in alumina.

Whenever a phase-rule diagram exhibits a maximum point, it always means that a chemical compound is formed between the two components, the melting point of this compound being the temperature corresponding to the maximum point and the composition of the compound being the abscissa of this point. Thus the diagram in Fig. 1 tells us that a chemical compound between magnesia and alumina containing 28 per cent of magnesia and 72 per cent of alumina and hence having the formula $Al_2O_3 \cdot MgO$ is formed and that this compound has a melting point of 2135°. If therefore we proceed to manufacture a refractory body by mixing together alumina and magnesia in the molal ratio 1 to 1 (*i. e.*, 28 per cent MgO and 72 per cent Al_2O_3 by weight), we can obtain a product which can be employed as a refractory at any temperature up to 2135° without the occurrence of any liquefaction. This magnesium aluminate is thus a somewhat better refractory than alumina alone, since it will stand a temperature 85° higher than pure alumina will. The diagram also shows that small errors in the composition of the body would not produce very serious results upon its refractory power since an excess of alumina lowers the liquefaction temperature only 210° and an excess of magnesia only 105°.

In addition to the increase in refractory power which can be obtained by adding to alumina enough magnesia to combine completely with it, certain other advantages are secured at the same time. We have mentioned above the difficulty of securing

a bond when manufacturing a refractory out of a single material; in fact, to manufacture a refractory of high mechanical strength out of pure alumina would require either long firing or a firing temperature close to the melting point, that is, a firing temperature of about 2000° which is scarcely practicable on an industrial scale, except with the aid of an electric furnace. By mixing together magnesia and alumina in the molal ratio of 1 to 1 and firing the resultant body, the compound, magnesium aluminate, will form, even at temperatures considerably below its melting point. The formation of this compound, which probably occurs through the gaseous phase owing to the mutual vaporization of the constituents in the pores of the body, usually results in a mass of small interlacing crystals and consequently gives a product of increased mechanical strength and toughness. The formation of this compound is accompanied by a large shrinkage during burning, and a consequent increase in density thus making it desirable to pre-calcine and grind part of the body mixture before shaping into the desired form.

The formation of such a compound between the two materials may also be expected to decrease greatly the volatility of both materials; to increase their resistance toward many chemical agencies and in some cases toward mechanical abrasion.

Still greater mechanical strength, as well as a diminished porosity and greater resistance toward mechanical abrasion than that which is produced by the chemical combination, referred to above, can of course be secured by adding to the body a material for the purpose of producing vitrification; but the increased strength and resistance obtained in this way can usually be secured only with the sacrifice of some of the refractory power. While it is not necessarily impossible to produce vitrification without loss of refractory power, extremely high firing temperatures would usually be required to obtain such a result.

The above conclusions may be summed up as follows: Any refractory body manufactured from alumina and magnesia will begin to liquefy at 1925° if it contains less than 71 per cent of alumina, and at 2030° if it contains more than 71 per cent of alumina. But a body having exactly the composition, 71 per cent alumina, will show no liquefaction whatever until a tem-

perature of 2135° is reached. The amount and composition of the liquid which is formed by heating a body composed of magnesia and alumina in *any given* proportions to *any given* temperature can be exactly calculated from the phase-rule diagram. Owing to the formation of a chemical compound between the constituents, decreased volatility with increased mechanical strength and resistance to abrasion and chemical action may be looked for. Numerous other examples illustrating the value of phase-rule diagrams in solving problems connected with the preparation and behavior of refractory materials might be cited.[1]

Among the substances which might properly be included in a comprehensive phase-rule investigation of refractory materials are the following: carbon, silica; the oxides of calcium, strontium, barium, magnesium, and beryllium; the oxides of alumina, iron, chromium and molybdenum; the oxides of titanium and zirconium; various rare earth oxides; and certain nitrides and carbides—such as boron nitride and silicon carbide. In addition to these substances, the oxides of the alkali metals should also be included because of their common occurrence as impurities in refractory raw materials and their powerful fluxing action, made use of in producing vitrification.

The total number of different systems which could be prepared by the combination of even 20 different components, up to and including four-component systems, is at least 6195, so that evidently the task of making anything like an exhaustive investigation covering all of the above materials would be enormous. It is evident therefore that a selection of the systems to be studied would be necessary, based upon the relative importance of these systems for practical purposes.

Most of the experimental work which has been required in order to complete the study of the few systems which have thus far been thoroughly investigated has been carried out in the Geophysical Laboratory of the Carnegie Institution of Washington. The phase-rule diagrams for the following systems have thus far been wholly or partially completed:

[1] See Sosman, *J. Ind. Eng. Chem.*, 8, 985 (1916); *Trans. Far. Soc.*, 12 (1917).

1. One-component systems: silica, alumina, magnesia, lime, carbon.

2. Two-component systems: silica–alumina, silica–magnesia, silica–lime, ferric oxide–lime, alumina–lime, alumina–magnesia, hematite–magnetite.

3. Three-component systems: silica–alumina–magnesia, silica–alumina–lime, silica–magnesia–lime, alumina–magnesia–lime.

4. Four-component systems (partial studies only): silica–alumina–magnesia–lime, silica–alumina–lime–soda, silica–alumina–potash–soda.

5. Five-component systems (partial studies only): silica–alumina–magnesia–lime–soda.

In view of the special knowledge and experience which the Geophysical Laboratory has obtained in dealing with the difficult problems connected with phase-rule investigations at high temperatures, it would certainly be advantageous if the direction and control of the work of a Division of Phase-Rule Investigations could be centered at this laboratory. In order to do this satisfactorily, it would probably be best to establish, if possible, a section in that laboratory for the special purpose of planning and conducting phase-rule investigations from the standpoint of the importance of these investigations to the subject of refractory materials. The work of the present staff of that laboratory is naturally inspired by geological interests and the phase-rule diagrams which have been worked out there are the result of the importance of these diagrams in their bearing upon geological and mineralogical problems rather than their importance in relation to refractory materials.

In addition to the actual experimental work which might be carried out at the Geophysical Laboratory in accordance with some coöperative arrangement, an advisory supervision and coordination of investigations along similar lines in other laboratories could to advantage be centered in the same institution which would thus become the headquarters for this Division.

In case the necessary arrangements could not be made with the Geophysical Laboratory, it would then be best, if possible, to center the work of this Division at one of the other institutions having well equipped ceramic laboratories. The Bureau of

Standards, the Bureau of Mines, the ceramic departments of several of the universities, and perhaps of certain private institutions should receive consideration in this connection.

10. **Division 3. Physical Constants.**—It would be the duty of this Division to compile all the available data concerning the physical, chemical and ceramic properties of refractory raw materials and manufactured products, to keep this compilation up-to-date in a readily accessible form, to promote the necessary investigations for increasing our knowledge of such properties, and to secure the coöperation of all investigators in this field to the end that experimental methods might be improved and standardized, and undesirable duplication avoided. Eventually this Division might desire to establish a laboratory of its own or arrange to have its work carried out by special arrangements with some existing laboratory, such for example as the Bureau of Standards.

11. **Division 4. Standard Methods for Testing Refractory Products.**—The function of this Division would be the development of standard methods for testing products in order that the results of such tests should indicate as clearly as possible the behavior which might be expected of each material under service conditions. The methods to be employed in testing a given material would naturally vary according to the use to which the material was to be put. The work of this Division should be correlated with that of the American Society for Testing Materials and the corresponding committees of the American Ceramic Society. For a time at least, the experimental investigations might be carried out by enlisting the coöperation of a number of different laboratories and the work might to advantage be centered at the Bureau of Standards.

The work of Divisions 3 and 4 deals with questions which are probably the most pressing ones at the present time. That is, an accurate and complete knowledge of the properties of refractory materials and products and the development of tests which will accurately depict the behavior of these materials under service conditions are very much needed.

12. **Division 5. Raw Materials Specifications.**—This Division would formulate the specifications to be met by each raw material employed in the manufacture of each type of refractory product. These specifications would cover such factors as chemical and mineralogical composition, crystallographic condition, texture, state of mechanical division, fusibility, and all the important ceramic properties such as plasticity, burning behavior, properties developed on burning, etc. The work of this Division might also be carried on for a time and perhaps permanently by coöperative arrangements with existing laboratories.

13. **Division 6. Manufacturing Methods.**—This Division would study the processes employed in manufacturing each type of refractory and recommend such changes as would result in improvements in quality, decreased cost of production and increased definition and standardization of product. Every effort should be made to design and manufacture refractories adapted to the special requirements of the furnace in which they are to be used. That is, insofar as commercially practicable, the refractory should in each instance be made for the furnace, not the furnace for the refractory. In an actual working organization, it would perhaps be desirable to unite Divisions 5 and 6.

14. **Division 7. Standard Specifications for Refractory Products.**—This Division, as well as Divisions 4 and 6 above, should probably be organized in sections, with a section for each industry requiring a special class of refractories. Among the most important users of refractory products which would be represented by sections in this Division, are:

1. The iron and steel industry.
2. The various non-ferrous metal industries.
3. The gas industry.
4. The by-product coke industries.
5. The glass industry.
6. The pottery and porcelain industries.
7. The brick, tile and sewer pipe industries.
8. The cement industry.
9. The various industries employing electric furnaces.
10. The enameling industries.

11. The great variety of chemical industries.

12. Power plants.

Each section would draw up the specifications which should be met by each type of refractory required in the corresponding industry.

The labors of this Division should be closely correlated with those of Divisions 3, 4 and 6, the work of which would, to a large degree, constitute the foundations upon which this Division would build. In a working organization, it would perhaps be desirable to unite Divisions 4 and 7.

15. **Division 8. The Engineering Division.**—The principal work of this Division would be in connection with the design and methods of operation of industrial kilns and furnaces, the purpose being to establish the best design of furnace or kiln and the most efficient method of operating the same for each particular industry or process employing high temperatures. This Division would perhaps be organized in sections corresponding to the various types of furnaces required. Owing to the large scale experiments involved in research in this field, it is clear that large expenditures would be required for carrying out any extensive research program.

16. **Division 9. Geology and Mining.**—The function of this Division would be to promote the extension of geological surveys looking toward the location and mapping of deposits of refractory raw materials, to investigate any other geological problems of importance to the subject, and to secure the further study and development and greater utilization of improved methods of mining, handling and preparing these materials. This Division should coöperate with the United States Geological Survey, the United States Bureau of Mines, the Association of American State Geologists, and the National Research Council, which organizations have recently formulated and are putting into effect a plan for a coöperative survey of the ceramic resources of the country. A description of this plan is contained in the proceedings of the Pittsburgh Conference.[1] This Division would coöperate closely with Division 5.

[1] *The Clayworker*, **69**, 759 (1918).

17. Division 10. Coördination and International Coöperation.—This Division would be composed of the chiefs of each of the other Divisions together with representatives of the National Research Council, and possibly of other organizations, under the presidency of the Scientific Director. It would be the duty of this Division to see that the necessary coördination was secured in the labors of each of the other Divisions and to promote any desirable international coöperation in refractory materials research. The headquarters of this Division might advantageously be located at Washington, and as far as international relations are concerned, the work of the Division could perhaps be most conveniently transacted through the international connections already established by the National Research Council.

This Division might also prepare a research census of all agencies engaged in refractories research and arrange to receive regular reports of progress.

An organization for coöperative research in refractory materials seems to be in process of formation in England and a report dealing with (*a*) Refractory materials required by the various industries; (*b*) The laboratory facilities available for refractory research in public institutions of Great Britain and Ireland; (*c*) The facilities existing in England for collecting and publishing information on refractory materials; (*d*) The problems calling for the most important action; and (*e*) The special requirements of each industry, was adopted by a conference held at London, in July, 1917, and has been published.

The ceramic industries of France have also recently formed an association known as the *Syndicat des Fabricants de Produits Ceramiques de France* and coöperative relations should be established with the refractories section of this association.

18. Financing the Research Organization.—An organization constituted in accordance with some such scheme as that outlined in the foregoing pages would require liberal financial provision for its operation. Ample financial resources for inaugurating the undertaking might be secured if each producer of refractory materials or products would contribute to a common fund an annual self-imposed tax of say 2/10 of 1 per cent of the annual

sales value of his output. In comparison with the tax which his business pays to state and nation, to fire, risk, and liability companies in the form of insurance premiums, and to legal counsel in the form of retaining fees, a research tax of 2/10 of 1 per cent is insignificant. Such a tax would provide an annual income of over $100,000 for a Refractories Research Corporation, and this income would automatically increase from year to year with the growth of the industry. If users of refractories, as well as producers, were admitted to the organization (and this would be highly desirable) the above estimated annual income of the Research Corporation could be increased by more than 50 per cent, by a similar rate of taxation on the annual purchase value of all refractories purchased by each user.

In this connection the following paragraphs from a recent address delivered by Dr. John Johnston before the American Zinc Institute are much to the point:

"Most large firms take out insurance of various kinds and make regular allowance for depreciation of plant and equipment; comparatively few make expenditures on research as a fixed charge on their business. And yet this insurance against ignorance is comparatively cheap; one large firm considers it certain that the amount gained directly from their research work—without taking into account the less tangible, though certain, benefits—has been *at least* ten times its cost. But to get the full benefit of such insurance it is necessary to go systematically into the fundamentals of the question, to ascertain precisely what is happening at each stage of the operation. The absolute necessity of such fundamental work is insisted upon by all of the big firms which have gone into systematic research work and found it profitable. This implies a fairly large expenditure which, though a relatively small matter for a large firm, would not be possible to a small firm because it would constitute too large a charge on its total annual product. But smaller units may organize to carry out research work coöperatively, and so gain equal, if not greater, advantage at a comparatively small cost to each individual unit."

"The National Canners Association some years ago established in Washington a laboratory to take up some of the difficulties of the canning industry; and this laboratory has been so suc-

cessful that it is now considered to be one of the assets of the industry. In Britain the plan of establishing a coöperative research organization is being considered by a number of industries; the most substantial progress has been made by the cotton industry, a provisional committee of which has worked out a scheme of procedure in considerable detail. They are establishing the British Cotton Research Association that will include as members, cotton-spinning and thread-making firms, manufacturers of cloth, lace and hosiery, bleachers, dyers, printers and finishers; it will conduct researches which include the study of the cotton plant at one end and the 'finishing' of the manufactured article at the other, and also encourage and improve the education of persons who are, or may be engaged in the industry. They have published a very interesting pamphlet[1] on 'Scientific Research in Relation to Cotton and the Cotton Industry,' in which a popular account of the matter is given. It would lead too far to go into the matter but this report brings out one important point deserving of mention here—namely, with respect to the cost of such research work to each member of the Association. On the basis that the Association would spend $250,000 a year on research work, it is shown that the cost to each member would be only about 10 per cent of his fire insurance premium, 25 per cent of the cost of health insurance, or about 20 per cent of the cost of employers' liability insurance. In other words, the scientific and technical health of the industry can be insured at a very small cost."

The organization of most of the Divisions would naturally be a gradual process dependent in each instance upon the finding of the right man to head the Division and the working out of a definite research program which would receive the approval of the Board of Trustees. Any attempt to create from the beginning a full fledged organization, such as that outlined above, would probably result in numerous disappointments. A not improbable eventual development of the work might be the establishment of a large central laboratory and testing station in which all of the research work of the Association would be carried out. The experience of several of the large corporations, such as the General

[1] Copies obtainable from the Secretary, 108 Deansgate, Manchester; (price nine pence).

Electric Company and the Eastman Kodak Company, and of the National Canners Association, has demonstrated the success of this method of conducting industrial research. Some of the obvious advantages of this concentrated form of organization over the distributed type of organization are (1) the opportunity for frequent personal conferences among the different types of experts on the staff; (2) the avoidance of duplication of equipment; (3) the saving of the time and energy of the Director. The first of these is probably the greatest virtue of the concentrated form of organization and is the one to which its great success is most largely due.

19. **The Practicability of Forming a Refractories Research Corporation.**—Whether and in how far it is feasible, under the industrial and economic conditions which exist in the refractories industries at the present time, to form a research corporation for the purpose of prosecuting coöperative research in this field could probably only finally be determined by attempting to form such an organization. Viewed purely from the scientific side, the field is a sufficiently homogenous one to make such an organization practicable and desirable, but viewed from the industrial and commercial side, this can hardly be said to be the case. The manufacturers themselves and many of the users of refractory materials are industrial competitors and this element of competition is alone sufficient to make the actual realization of any such plan as that proposed here extremely difficult, if not quite impossible. Industrially and commercially the field is a very heterogeneous one, involving many diverse and conflicting interests. Some of the larger manufacturers already have their own research laboratories for dealing with the problems peculiar to their own product, and the first interest of each manufacturer is naturally the well being of his own business rather than the development and perfection of our knowledge of refracto y materials in general.

The preparation of any research budget to be defrayed by appropriations from a common fund contributed to by *all* manufacturers and users of refractories would present almost insuperable difficulties. Perhaps the only Divisions in which anything

like unanimous agreement as to expenditures could be secured would be Divisions 1 and 9. In the case of the other Divisions it would seem that a budget could be agreed upon only if the expenses of the different undertakings in each Division were defrayed by those manufacturers and users to whom the results of these particular investigations would be of direct and immediate value. For example, to take an extreme case, one could hardly expect that the manufacturers of clay refractories would be especially interested in helping to defray the expenses of an investigation of boron nitride as a refractory. Certainly if any part of a common fund were used in this way, any contribution of the manufacturers of clay refractories towards such investigations would be a purely altruistic one. Furthermore, any common fund created by contributions based upon the sales and purchase value of products would in large part be made up of contributions from manufacturers and users of a comparatively small number of types of refractories, since most of the tonnage of refractories used in modern industry consists of clay and silica materials, and these contributors would naturally be primarily interested only in investigations dealing with these particular classes of refractories. For the same reasons any considerable expenditure for the work of Division 2, for example, could hardly be expected from other than altruistic motives, since the phase-rule diagrams for the refractory materials most largely used and for which the necessary tonnage of raw materials is in sight have already been worked out and hence the development of any refractory which would to any considerable degree replace any of these materials would only be commercially possible in case it had a much longer life under service conditions than the types of refractories now employed.

Moreover, it could hardly be expected that the manufacturers of a given type of refractory would be interested in promoting an investigation which might conceivably result in the perfection of a product of sufficient commercial practicability to destroy or to greatly injure their own business, however desirable from a national viewpoint such a result might be. Certainly such investigations could be undertaken only if adequate provisions for safe-guarding the financial interests of all members against

such possibilities as the above could be worked out. A single industrial corporation, such for example as the General Electric Company, can undertake investigations leading to the development of products which might require the scrapping of manufacturing facilities representing large investments, but for an association of more or less competitive interests to undertake any such investigations on a coöperative basis has obviously great difficulties, especially if both manufacturers and users are included in the organization. These difficulties would, it is true, be greatly diminished if the association included only users of refractory materials, and for this reason it may be that the country will eventually have to look to the consumers of refractories for the prosecution in a large way of systematic research in this field.

It seems clear to the writer that any such organization as the "ideal" one presented in the preceding pages could scarcely be developed even eventually except by private or governmental endowment or by the actual industrial amalgamation of all or the greater part of the refractories interests of the country. In view of these facts it may be that the above survey of the scientific aspects of the subject has no practical value at the present time, but if it serves only to promote discussion, to arouse further interest in research in this field, and to bring about any further degree of coöperation, its purpose will be fulfilled. Some type of organization for accomplishing at least a portion of the work outlined above ought to be feasible. Perhaps several more or less independent organizations, instead of one, would be required, or perhaps an association of research laboratories engaged in refractories research might be able to perfect some practicable scheme of coöperation. Certainly, viewed solely from a national economic viewpoint, the stimulation of research in this field is greatly needed, and in addition to supporting the investigation of those problems which seem most urgent from the "practical" viewpoint, it is to be hoped that to some degree at least manufacturers and users of refractories will be willing to make some contributions from more purely altruistic motives; that is, that they will feel ready to take some share in the promotion of all classes of investigations in their field, even though they see no possibility of direct or immediate financial return to themselves.

Such contribution might take the form of direct financial assistance or the form of supporting the efforts of federal, state and endowed institutions to secure larger appropriations for carrying out some of the more fundamental scientific work, even though some of this work may seem to be largely of the type ordinarily classed as "pure science," and perhaps appear to be more or less visionary or "impractical." Certainly the history of the development of science and industry has repeatedly demonstrated the enormous ultimate value of research work of this character.

THE RELATIVE ACTION OF ACIDS ON ENAMELS—III.

By E. P. Poste.

Introduction.

The action of acids on enamel surfaces is a matter of continued interest to those engaged in the various phases of enamel technology as well as to anyone who is engaged in the manufacture of a glass or a silicious protective coating which is to withstand the attack of acids. The importance of the subject has brought about the need for some standard test for acid resistance and one which can be generally accepted and by means of which wares can be compared by different parties in such a manner as to secure accurately comparable results.

The Sub-Committee on Enamels of the Committee on Standards of the American Ceramic Society has been assigned a task of this kind and, while the work is not sufficiently advanced to take the form of a report, some preliminary efforts which throw considerable additional light on the general subject under consideration have been made. Before treating in a detailed way the results of the present work it will be well to refer briefly to records of previous investigations.

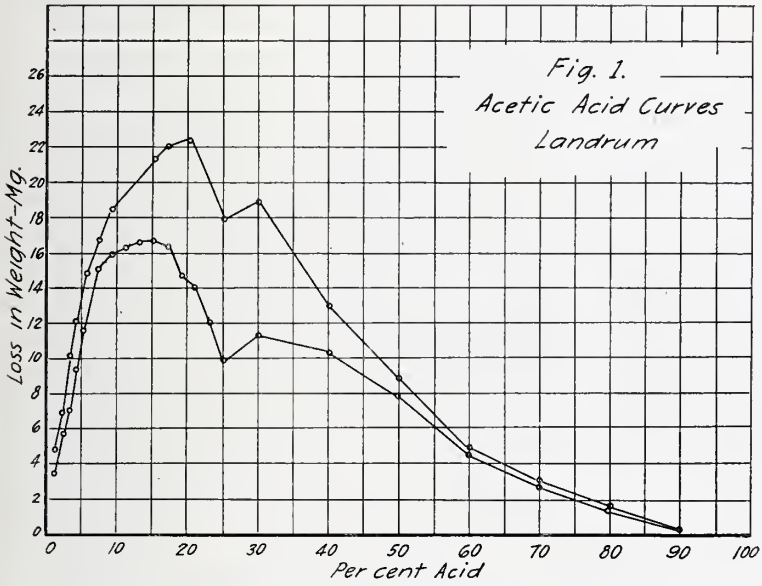
Previous Investigations.

Definite information as to the actual nature of the action of an acid on an enamel has been recorded by R. D. Landrum.[1] His method of testing was to evaporate to dryness various initial concentrations of acetic acid in small enameled basins and to determine the loss in weight due to the action of the acid under these conditions. The curves which he obtained from two series of such tests are shown in Fig. 1. Landrum's general conclusion was that the maximum activity took place in the case of an acid approximately 20 per cent in original strength and that the action rapidly diminished as the initial acid approached a concentrated

[1] *Trans. Am. Ceram. Soc.*, **13**, 494–501 (1911).

solution. He noted, at about 25 per cent, a definite irregularity in the curve. This he was able to check upon repeated trials.

The next quantitative data reported were published by the writer.[1] The method used was to expose a sample of standard enamel frit to the various strengths of acids under standard conditions and to determine the loss in weight. It was demonstrated in general that the maximum activity of an acid on an enamel took place at concentrations of from 10 to 20 per cent. This fact was re-



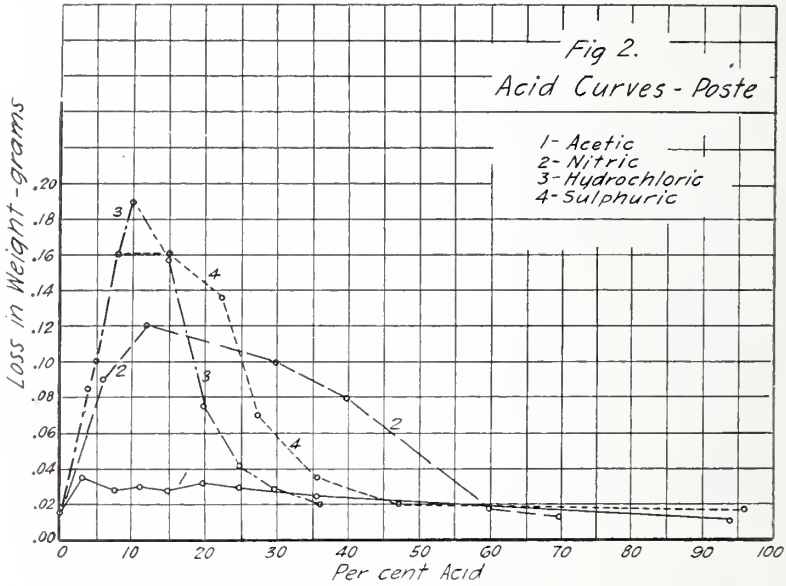
demonstrated relative to acetic acid and was extended to include sulphuric, nitric and hydrochloric acids. The results are shown in Fig. 2.

A second paper by the present author[2] was fundamentally an answer to the discussion of the first paper and indicated the effect of the various possible irregularities in the method—which as a whole has been rather severely criticised as not being of particular value in the determination of the acid resistance of finished enamels.

[1] E. P. Poste, *Trans. Am. Ceram. Soc.*, **17**, 137–149 (1915).

[2] E. P. Poste, *Ibid.*, **18**, 570–574 (1916).

An investigation has also been reported by Leon J. Frost[1] who presented data resulting from a study of the action of acetic acid solutions of varying strengths on finished enamel surfaces. The method used was essentially the same as that which has been used by Landrum—varying only in the manner of heating. The curve which Frost obtained is shown in Fig. 3. He confirms the conclusions already drawn and, besides checking in a general



way the irregularity in the curve as noted by Landrum, he reports several other irregularities occurring at lower concentrations.

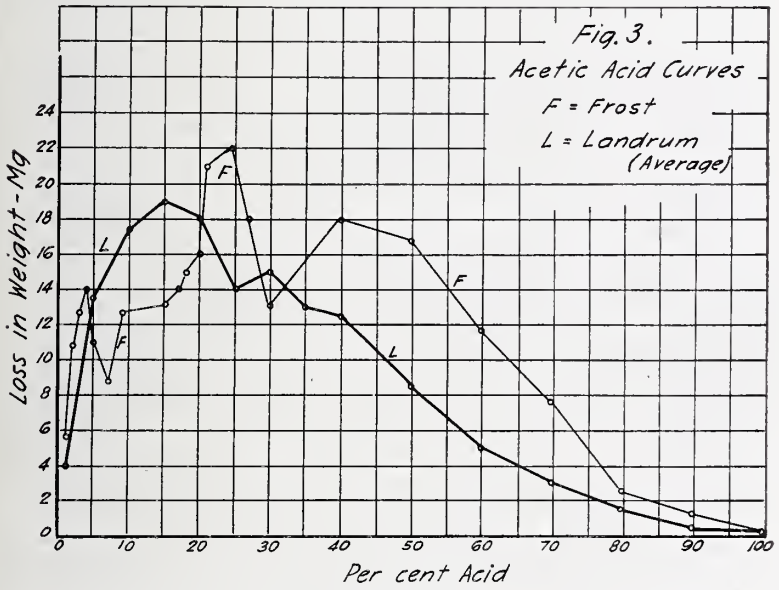
Present Investigation.

The work of the Sub-Committee on Enamels has included a review of the status of the situation as outlined above and a consideration of the fundamentals upon which an acceptable test should be based. This has been followed by certain experimental work. As one of the fundamentals, the members of the Committee have agreed that an acid to be used in a standard test should be one which can be obtained at the average drug store and made

[1] This Journal, 1, 422 (1918).

up to a given strength by the practical man without introducing errors which would exceed a reasonable limit of experimental error in the final results of the test.

After considerable discussion of this phase of the question it appeared that a material which could be bought in the solid form and added to a certain amount of water, thus producing a standard solution of the pre-determined strength, would be much preferable to an acid purchased in the liquid condition which would be much more liable to variation in strength. It would of course



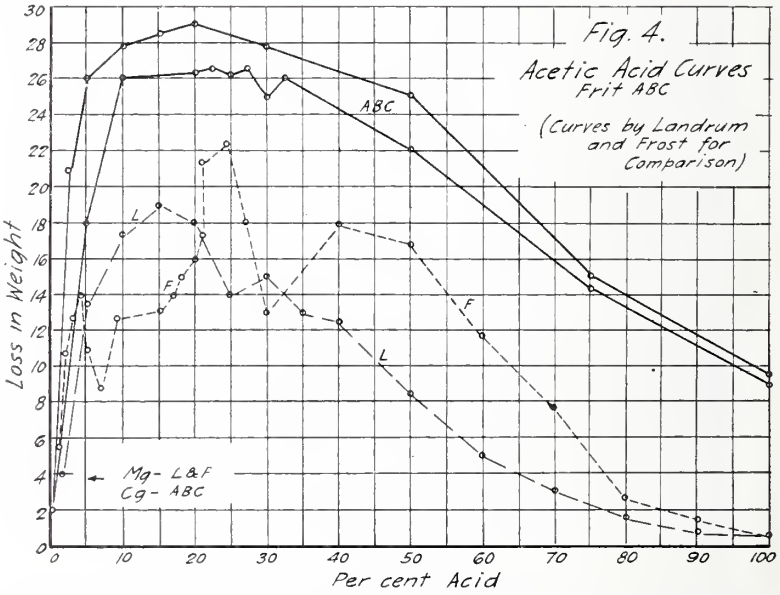
be pre-requisite that such a solid reagent as obtained from normal stock should be reasonably uniform in its composition. The data presented in this paper are those which have resulted from studies along these lines.

Enameled cooking ware is very often called upon to resist the action of citric and tartaric acids and as these promised to be of value as a basis for a standard test they were chosen as a basis of study. The preliminary work of the Committee to date has included a determination of the characteristic behavior

of these acids towards enamels. Their stability under storage conditions has also been investigated.

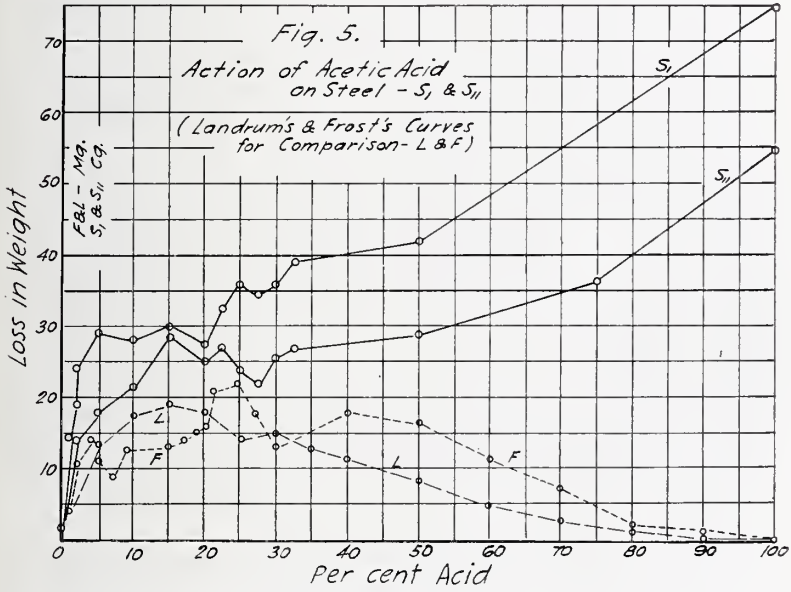
As a basis of such work five frits were used. They were designated as Ex. 1, Ex. 2, A, B and C. Ex. 1 and Ex. 2 were two experimental blue frits of low acid resistance as compared with cooking-ware frits. A, B and C were three commercial white frits submitted to the Committee by three enameling companies. A mixture of equal parts of these was designated as ABC.

Action of Acetic Acid.—In an effort to correlate the previous data on acetic acid with the action of acetic acid on the enamels



under consideration, several series of tests were made—using a mixture of equal parts of the above mentioned frits (A, B and C). A frit tested under standard conditions gave the curve shown in Fig. 4, in which are also reproduced the curves of Landrum and Frost. It will be noted that the general nature of the curves is quite uniform although the action of the acid at higher concentrations appears to have been relatively more severe than the action on the enamels used in previous work.

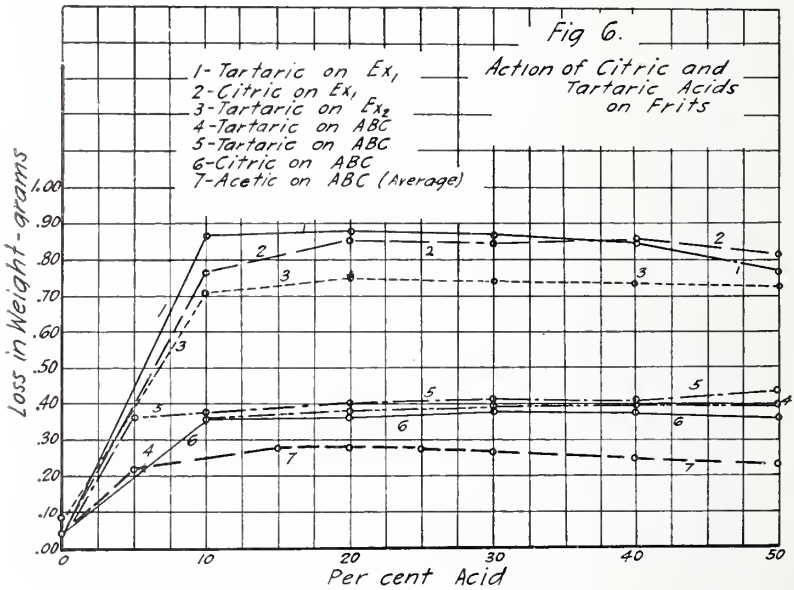
At this point it seemed well to note the action of acetic acid on freshly cleaned steel surfaces and two series of tests were run on small basins, the losses in weight of which were determined gravimetrically. These basins were exposed to equal amounts of various strengths of acetic acid at laboratory temperatures for 24 hours. The results are plotted in Fig. 5, the two curves representing two different tests, presented separately rather than as one curve representing the average, in order to show their similar nature. The numerical difference was due to a higher average temperature in one case than in the other. It is interesting



to note the parallel irregularities up to 30 per cent which suggest a relation to the irregularities obtained by Frost and Landrum. Their curves have been reproduced on this figure for reference in connection with this point. It will further be noted that the acetic acid at higher concentrations acted very severely on the steel as compared with the more dilute solutions—which is in accordance with the generally accepted fact that the acetic acid has a particularly strong corrosive action on steel, even in the absence of water.

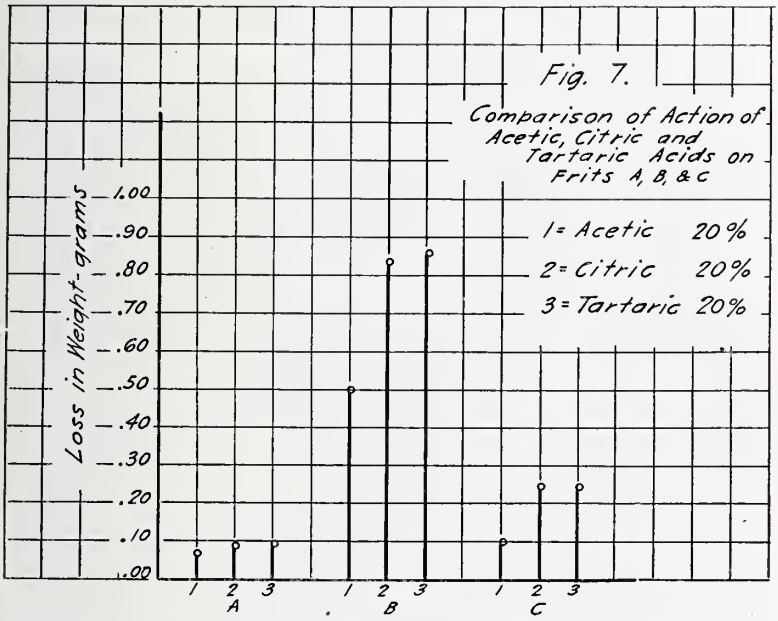
Action of Citric and Tartaric Acids.—The investigation as to the action of tartaric acid included tests on the three frits, Ex. 1, Ex. 2 and ABC, the results of which are shown in Fig. 6. Similar concentrations of citric acid were used on the frits Ex. 1 and ABC, the results of which are also shown in Fig. 6 as well as the acetic acid curve for ABC.

Two general observations may be made. One is the fact that enamels of different acid resistance behave quite differently when subjected to the action of either citric or tartaric acid. This fact is very evident from the characteristic difference between the curves for frits Ex. 1 and Ex. 2 and those for frit ABC. It



is also very apparent that the relative action of citric and tartaric acids on the combined frit ABC is not materially different and that in concentrations, from 10 per cent up to saturation, the severity of the action is quite constant. It would seem that the above tests have established in a general way the nature of the action of citric and tartaric acids on the particular frits used and that the activity of a 20 per cent solution was quite high, without any very radical changes due to slight differences in acid concentrations in either direction being in evidence.

The next test was to run separately on 20 per cent acetic, tartaric and citric acids, each of the three frits which had been previously used in the mixture ABC. The results of these tests are shown in Fig. 7 from which it will be seen that frit A is of relatively high acid resistance, being practically equally resistant to 20 per cent solutions of acetic, citric and tartaric acids. Frit B is much less resistant than frit A and considerably less resistant to tartaric and citric acid than to acetic acid. Frit C is quite

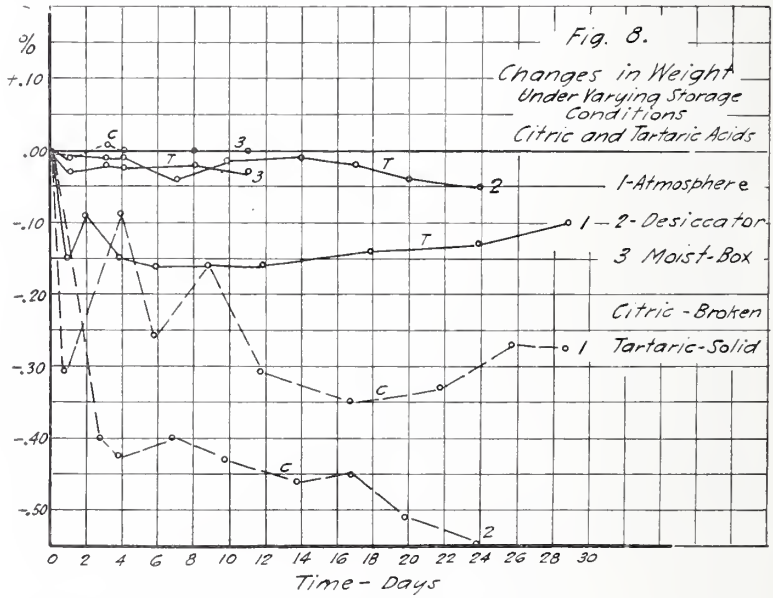


resistant to acetic acid and much less resistant to tartaric and citric acids.

Variation in Composition of Citric and Tartaric Acids.—To determine the possibility of obtaining either citric or tartaric acid which would be quite uniform in strength, two lines of investigation were carried out. The first method was to expose 5-gram samples of tartaric and citric acids to varying atmospheric conditions over a sufficiently long period of time to determine any tendency to change in weight; the second was to procure from local drug stores several samples of citric and tartaric acids

and to compare their compositions, assuming that they may have been subject to variations in storage conditions.

The results of the former tests are shown in Fig. 8. The curves marked 1 show on a percentage basis the change in weight of citric and tartaric acids when exposed to the laboratory atmosphere during the month of December, at which time the room was maintained at a temperature approximating 20° C, but owing to various operations going on in the room the humidity was

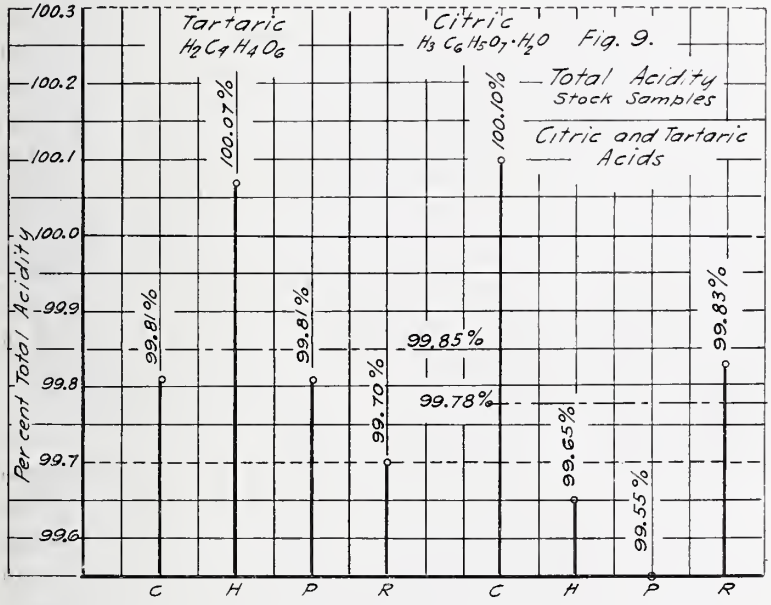


no doubt relatively high. It will be seen that the citric acid lost weight up to about 0.3 per cent, beyond which no very material change took place, and that the tartaric acid lost weight to about 0.15 per cent—beyond which it was reasonably uniform.

The curves marked 2 (Fig. 8) were plotted from the results of similar tests made upon samples which were placed in a desiccator over calcium chloride. It will be noted that the citric acid lost weight quite rapidly to about 0.4 per cent and continued to lose somewhat gradually from that point on while tartaric acid lost but very slightly, the maximum being about 0.05 per

cent. The curves marked 3 were plotted from the results of a test in which the two materials were exposed in a moist box to a humidity approximating 85 per cent (as determined by the wet- and dry-bulb thermometer method) at temperatures ranging from 15° to 20° C. It is quite apparent that there was no material change in weight, the tartaric acid losing slightly more than the citric acid but not over 0.03 per cent during the period of the test.

From these tests it would seem that tartaric acid is much less subject to changes, due to varying storage conditions, than is



citric acid. This fact is quite readily explained by a consideration of the chemical composition of these two materials. The formula of tartaric acid is $H_2C_4H_4O_6$ and that of citric acid $H_3C_6H_5O_7 \cdot H_2O$. In other words, the tartaric acid molecule does not contain water of crystallization while the citric acid molecule does have water of crystallization which is subject to partial loss when the acid is exposed to an atmosphere of low humidity.

The analytical data relative to the various samples of citric and tartaric acid are shown in Fig. 9 in which is indicated the

variations in total acid content of the various samples obtained. It will be noted that the total acidity figure for tartaric acid ranged from 99.7 to 100.07 per cent, giving an average of 99.85 per cent, and that similar values for citric acid ranged from 99.55 to 100.10 per cent, giving an average of 99.78 per cent. These figures can only be taken to mean that both of these materials as tested conform to the theoretical composition within the limits of the analytical method used—which cannot be taken as accurate within plus or minus 0.3 per cent, the upper and lower limits of such error being designated by the two dotted lines at 100.3 per cent and 99.7 per cent, respectively. It will be noted that the average values for citric acid are slightly lower than those for tartaric acid but no significance can be attached to this fact in view of the slight difference as compared with the experimental error.

Conclusions.

The above review and further investigation relative to the action of acetic acid on enamels demonstrate the fact that the dilute acids are more active than the concentrated—the maximum being in the neighborhood of 20 per cent. The action of acetic acid in the neighborhood of its maximum activity on an enamel is subject to various irregularities. This has also been found true of the action of acetic acid on steel. This characteristic behavior of acetic acid would indicate that it is not desirable as an acid for a standard test in view of the fact that slight variations from a given strength would cause quite material differences in the activity of the acid on an enamel. A further objection to acetic acid would be the fact that it is a liquid which, when obtained from the average drug store, might vary quite materially in strength and hence analytical methods might be necessary in the preparation of a standard solution.

Owing to the fact that citric and tartaric acids are obtained in the solid form and are generally considered quite pure, they have been investigated and the following points brought out: First, at a given concentration citric and tartaric acids are more active on an enamel than is acetic acid and hence they give a more definite result in a given time. Second, the relative action of citric and tartaric acids at given concentrations appears to be about equal.

Third, the general shape of the curves for citric and tartaric acids is such as to render slight errors in concentration above 10 per cent of minor importance in terms of the difference in activity, *i. e.*, the curves from 10 per cent to 50 per cent are practically horizontal straight lines.

As a bearing on the relative merits of citric and tartaric acids for the proposed test, a study of the susceptibility of these two materials to changes in weight—due to varying storage conditions—has indicated very definitely that tartaric acid is much more stable than citric acid. In order to determine the probable variation on samples of commercial materials, four samples of each (citric and tartaric) obtained from stock and analyzed indicate that the variations—due to changes in moisture content which are liable to occur—are very slight, as a matter of fact they are within the limits of error of the analytical methods used, and at the same time so slight as to make practically no material difference on the action of the acid on the enamel, brought about by the slight variation in strength of a standard solution resulting from these irregularities.

We realize that the work which has been done along this line has been preliminary and that it has brought out several possible conditions which should be investigated further. Besides covering these points, the proposed line of procedure involves an effort to extend the tests to include the use of the proposed acids on actual enamel surfaces.

We will be very pleased to receive suggestions as to the relative desirability of using citric or tartaric acids in a standard test—in view of the general conclusion that either one of these acids would be appropriate so far as revealed by the data herein presented.

Elyria Enameled Products Co.,
Elyria, Ohio.

COMPRESSIVE STRENGTH OF CEMENT-LIME MORTARS.[1]

By F. A. Kirkpatrick and W. B. Orange.

Introduction.

Cement mortars have been made the subject of many investigations, as a result of which the laws governing the strength of such mortars are quite well understood.

It is not known, however, whether these laws hold when hydrated lime is included as a constituent of cement mortar. Very few investigations have been made of cement-lime mortars and none of them has had as its object the determination of the laws governing strength.

Macgregor[2] has determined the strength of a number of cement-lime mortars. He determined also the strength and moduli of elasticity of brick piers using such mortars. His principal conclusion was that the substitution by volume of hydrated lime in a 1 : 3 cement mortar caused decreases in strength for all amounts used. The strongest brick piers, however, were those made with mortars in which 10 to 50 per cent of the cement by volume had been replaced by hydrated lime.

Wig and Bates[3] determined the effect of the addition of waterproofing powders upon the compressive strength of 1 : 4, 1 : 6, and 1 : 8 portland cement mortars. Their conclusion in regard to strength was as follows: "The addition of the inert void fillers to mortars, as used in these tests, up to 20 per cent of the volume of cement, increases the compressive strength." The materials added included clay, feldspar, sand, hydrated lime and mixtures of these materials with organic substances.

A number of properties of cement-lime mortars were investi-

[1] By permission of the Director, Bureau of Standards.

[2] Bulletin J. of the Hydrated Lime Bureau, Pittsburgh, Pa., June 1, 1916.

[3] "Tests of the Absorption and Permeable Properties of Portland Cement Mortars and Concretes," Bureau of Standards, *Technologic Paper* 3.

gated by Emley.[1] The object of the investigation was stated to be twofold; first, to show the relations between properties of the mortars and the proportions of the ingredients; second, to illustrate the value of the triaxial diagram as a means of studying the properties of mortars.

The object of the present investigation was to determine what factors exert the greatest control over the strength of cement-lime mortars, to express the manner of control by a mathematical formula, and to indicate practical applications of the results.

Methods.

The following is a description of the materials used and of the methods employed in the experimental work.

Allegheny River sand, weighing 100 pounds per cubic foot and having a specific gravity of 2.65, was used. This material was of the following granulometric composition:

Per cent. passing sieve.	Mesh of sieve.	Size of opening. Inch.
100.0	20	0.034
88.2	30	0.0198
45.3	40	0.0150
10.5	60	0.0087
5.1	80	0.0068
3.1	100	0.0055
1.4	200	0.0029

A Western Pennsylvania portland cement was used. It had a specific gravity of 3.13 and was assumed to weigh 100 pounds per cubic foot.

An Indiana high calcium hydrated lime was employed. It had a specific gravity of 2.32 and weighed 40 pounds per cubic foot.

The mixtures were compounded upon the basis of the bulk volumes of the materials as given above, the water also being measured by volume. The compositions were chosen by means of the triaxial diagram as shown in Fig. 1. The figures shown at 5 per cent intervals are the designating numbers used for the compositions chosen. It should be noted that Figs. 1 and 4

[1] "Properties of Cement-Lime-Sand Mortars," *Proc. A. S. T. M.*, **17**, Part II, 261 (1917).

represent only one-quarter of the complete triaxial diagram but include most of the practical mortars.

The mortars were compounded carefully. The dry materials were weighed accurately and mixed thoroughly upon a mixing cloth. Water was then run in from a burette until the mixture

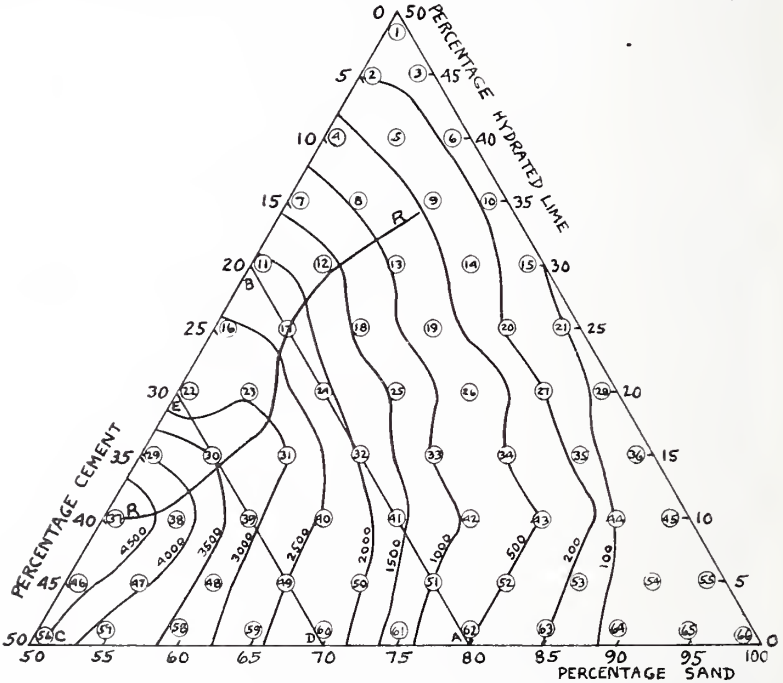


FIG. 1.—Compressive strength, pounds per square inch, at age of 28 days' air storage.

attained normal consistency as determined by the ball method. This method was carried out as follows:

A portion of the mortar was molded in the hands to the shape of a sphere, approximately two inches in diameter. When let fall upon a hard surface from a height of two feet, the ball must deform to a mass having a thickness of half its initial diameter. The mortar is then said to be of normal consistency. This determination could not be made for the very sandy mortars. These were brought as nearly to normal consistency as could be

judged by working them by hand and trowel. The method was hence somewhat unsatisfactory. The mortars were, of course, much less deformable than those used by the mason. It is suggested that for future investigations along this line, mortars of the consistency used by the mason be employed. Determination of the consistency of such mortars could be made more accurately. One method which has been attempted successfully for this purpose is that of filling a cylindrical mold with the mortar, pulling the mold up vertically, and measuring the height and diameter of the deformed mass of mortar. This method

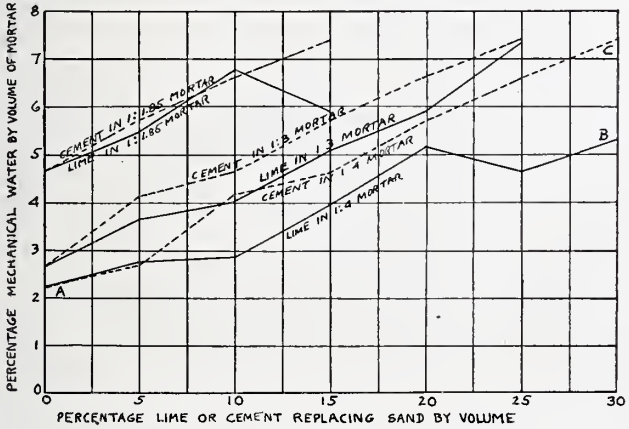


FIG. 2.—Mechanical water in mortars at age of 28 days' air storage. Proportions by volume.

can be used even for the more sandy mortars if they are brought to the soft consistency required by the mason.

As will be shown later, the maintenance of a standard consistency (measured in terms of deformability) in a series of mortars whose strength is to be studied is of practical value only—since strength does not vary with proportions of materials in the same manner as does consistency.

For each mortar composition chosen there were molded, in the customary cement gang-molds, six two-inch cubes. This was accomplished by forcing into the mold with the hands a large lump of the mortar which had been kneaded and rolled into a compact mass. The mold was then reversed and the other side

of the specimen subjected to pressure by the hands and more mortar used if necessary to fill the mold. The mold was weighed with and without the mortar, thus giving data for the calculation of weight per unit volume. As the results will show, the volume of the gang-mold remained sufficiently constant for our purposes. The specimens were allowed to stand in the molds in a moist place over night. They were then removed, three placed in water and three in air storage in the laboratory.

At the end of a 28-day storage period, the specimens were broken

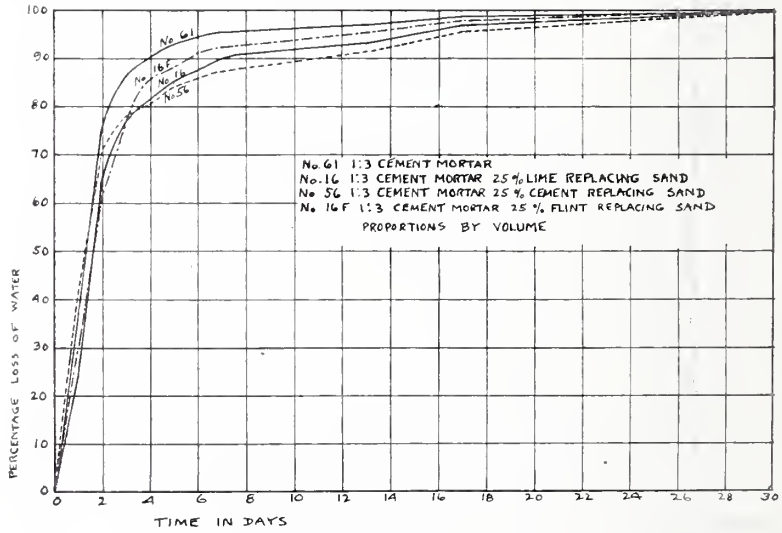


FIG. 3.—Rate of drying of mortars in air storage.

for crushing strength in a 10,000 pound hand-power Olsen testing machine. The broken pieces were used for determination of porosity by the usual method.[1] This consists in weighing the specimens after drying at 110° C, immersing them in water under a vacuum for at least 12 hours, then weighing wet, and weighing again suspended in water.

Discussion of Results.

The numerical results are presented in Table 1. Column No. 1 of this table gives the designation number of the mortars.

[1] R. C. Purdy, Illinois Geol. Surv., *Bull.* 9.

Columns Nos. 2 to 5 give the composition by bulk volume. Nos. 6 to 10 give numerical values representing the factors which govern the strength more or less accurately. Columns 11 and 12 give the compressive strength for air storage and water storage specimens.

Complete results for water storage mortars only will be presented

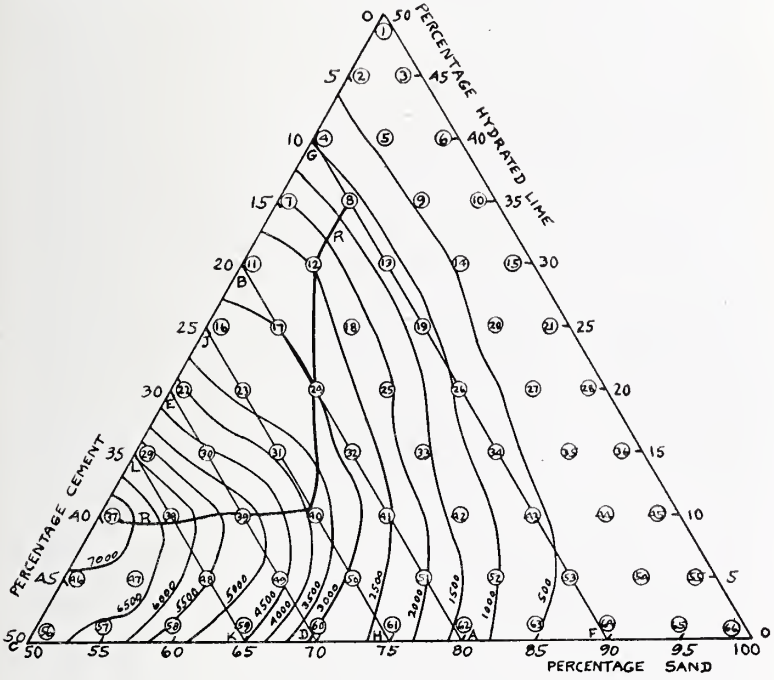


FIG. 4.—Compressive strength, pounds per square inch, at age of 28 days' water storage.

diagrammatically. There are two main reasons for this procedure: first, the systematic changes in properties are practically the same for either kind of storage; second, there is a maximum development of strength in water storage for most of the mortars.

Relations between Strength and Dry Bulk Composition.

The relations between strength and dry bulk composition of air storage mortars are shown in Fig. 1 and of water storage

TABLE I.

No. (1)	Composition by bulk volume.				Density. (6)	Cement density product. (7)	Water ratio. (8)	Porosity, air storage. (9)	Porosity, water storage. (10)	Compressive strength, air storage. (11)	Compressive strength, water storage. (12)
	Sand. (2)	Cement. (3)	Lime. (4)	Water. (5)							
1.....	50	0	50	23.6	...	....	...	..	..	145	..
2.....	45	5	45	24.0	0.656	0.239	4.800	..	34.0	195	175
3.....	55	0	45	23.2	...	....	...	..	..	160	..
4.....	50	10	40	23.6	0.693	0.0494	2.360	29.5	28.0	700	925
5.....	55	5	40	23.2	0.676	0.0245	4.650	..	29.3	300	290
6.....	60	0	40	22.8	...	....	...	..	..	130	..
7.....	50	15	35	23.2	0.666	0.0726	1.550	27.8	25.5	1385	2095
8.....	55	10	35	22.8	0.702	0.0524	2.280	27.6	25.9	950	1250
9.....	60	5	35	22.1	0.713	0.0263	4.420	..	27.2	405	370
10.....	65	0	35	24.0	...	....	...	..	..	110	..
11.....	50	20	30	21.6	0.691	0.1010	1.080	25.2	23.7	2185	2975
12.....	55	15	30	21.2	0.715	0.0801	1.413	26.2	24.5	1670	2495
13.....	60	10	30	21.6	0.711	0.0522	2.160	25.5	26.2	950	1175
14.....	65	5	30	21.6	0.704	0.0254	4.320	..	26.6	280	420
15.....	70	0	30	23.6	...	....	...	..	..	100	..
16.....	50	25	25	23.2	0.681	0.1191	0.928	26.4	26.4	2580	3070
17.....	55	20	25	22.0	0.699	0.0992	1.100	25.5	24.6	2320	3030
18.....	60	15	25	22.4	0.711	0.0769	1.493	26.2	24.2	1365	2370
19.....	65	10	25	22.4	0.710	0.0503	2.240	26.4	25.3	675	1130
20.....	70	5	25	24.8	0.674	0.0235	4.960	..	..	180	200
21.....	75	0	25	22.4	...	....	...	..	..	75	..

22.....	50	30	20	23.2	0.687	0.1415	0.775	25.5	25.1	2585	4140
23.....	55	25	20	22.8	0.700	0.1218	0.910	24.6	25.1	2925	3145
24.....	60	20	20	23.2	0.710	0.0994	1.160	25.5	26.3	2145	3060
25.....	65	15	20	22.4	0.711	0.0741	1.490	26.8	25.8	1415	2115
26.....	70	10	20	23.6	0.694	0.0465	2.360	..	27.4	765	890
27.....	75	5	20	24.0	0.690	0.0228	4.800	..	..	205	210
28.....	80	0	20	24.8	...	....	...	..	..	50	..
29.....	50	35	15	22.1	0.712	0.1738	0.631	23.0	18.6	4110	5970
30.....	55	30	15	21.7	0.716	0.1490	0.723	23.0	20.1	3480	4565
31.....	60	25	15	22.1	0.716	0.1232	0.882	24.2	23.5	3110	3885
32.....	65	20	15	23.2	0.700	0.0931	1.160	25.5	25.6	1925	2750
33.....	70	15	15	24.0	0.688	0.0670	1.600	27.6	27.2	970	1560
34.....	75	10	15	23.2	0.689	0.0445	2.320	29.5	29.0	485	650
35.....	80	5	15	24.8	0.668	0.0200	4.960	..	30.2	150	140
36.....	85	0	15	..	...	....	...	..	..	..	..
37.....	50	40	10	22.8	0.731	0.2100	0.570	22.9	17.9	5090	7540
38.....	55	35	10	22.8	0.726	0.1760	0.650	23.1	14.8	4340	6440
39.....	60	30	10	22.8	0.715	0.1502	0.760	24.0	18.8	3105	5300
40.....	65	25	10	22.8	0.724	0.1223	0.911	25.0	21.0	2540	3910
41.....	70	20	10	22.8	0.693	0.0794	1.140	25.9	23.9	1720	2570
42.....	75	15	10	23.6	0.705	0.0684	1.572	27.4	26.3	910	1355
43.....	80	10	10	23.2	0.685	0.0429	2.320	30.0	30.5	485	585
44.....	85	5	10	24.7	0.641	0.0186	4.950	..	29.2	100	120
45.....	90	0	10	..	...	....	...	..	..	..	..
46.....	50	45	5	23.6	0.708	0.2113	0.525	23.2	20.8	4800	6920
47.....	55	40	5	22.7	0.717	0.1918	0.570	23.1	20.5	4150	6790
48.....	60	35	5	22.4	0.709	0.1624	0.640	24.4	21.8	3210	5540
49.....	65	30	5	22.0	0.717	0.1390	0.734	24.7	22.2	2490	4810

TABLE I—(*Continued*).

No. (1).	Composition by bulk volume.				Density. (6)	Cement density product. (7)	Water ratio. (8)	Porosity, air storage. (9)	Porosity, water storage. (10)	Compressive strength, air storage. (11)	Compressive strength, water storage. (12)
	Sand. (2)	Cement. (3)	Lime. (4)	Water. (5).							
50.....	70	25	5	23.7	0.711	0.1150	0.947	25.2	24.6	2110	2800
51.....	75	20	5	24.8	0.700	0.0882	1.24	27.4	25.3	940	2040
52.....	80	15	5	26.8	0.678	0.0616	1.788	29.5	28.5	465	1015
53.....	85	10	5	27.2	0.646	0.0370	2.720	23.3	30.6	150	375
54.....	90	5	5	30.0	0.628	0.0186	6.000	..	..	30	60
55.....	95	0	5	..	...	....	...	..	..	..	..
56.....	50	50	0	24.0	0.715	0.2328	0.480	21.4	16.3	4760	6830
57.....	55	45	0	23.6	0.707	0.2050	0.525	23.4	17.7	3580	6260
58.....	60	40	0	22.8	0.707	0.1837	0.570	22.1	18.8	3475	5180
59.....	65	35	0	23.0	0.706	0.1560	0.658	24.2	20.3	2545	4380
60.....	70	30	0	24.3	0.699	0.1312	0.810	26.1	23.6	2330	2850
61.....	75	25	0	23.2	0.682	0.1023	0.925	27.2	25.8	1225	2370
62.....	80	20	0	26.0	0.657	0.0706	1.300	30.4	27.6	440	1330
63.....	85	15	0	28.0	0.643	0.0544	1.867	..	32.1	200	500
64.....	90	10	0	32.0	0.618	0.0328	3.200	..	..	65	125
65.....	95	5	0	32.4	0.621	0.0164	6.88	..	..	15	25
66.....	100	0	0	..	...	....	...	..	..	..	..

mortars in Fig. 4. The percentage compositions of the mixtures may be read from these diagrams or found by reference to the proper designating number in Table 1.

The triaxial method of representation is unsatisfactory for the purpose of a theoretical study of the properties of cement-lime mortars. Since there are four constituents in these mortars, it would be necessary to produce four triaxial diagrams in order to provide for all the possible combinations of three constituents each. The writers have found that these four diagrams give little satisfactory theoretical information.

Since the contour or iso-strength lines of Fig. 4 appear to run their courses in a uniform manner, it would seem that there must be a definite law governing their directions. As will be shown later, such a law exists and depends fundamentally upon the density of the mortars. Since both strength and density cannot be represented at the same time in the triaxial diagram of Fig. 4, the operation of the law governing strength cannot be satisfactorily observed by means of this diagram.

Effect of Replacing Sand by Cement or Lime.

Before taking up the theoretical discussion it is desired to consider the practical significance of the results presented in Figs. 1 and 4.

The claim is often made, by advocates of the use of hydrated lime in mortars and concretes, that the lime takes up water which it retains and later gives up to the cement for hydration, thus causing increase in strength. In Fig. 2 is shown diagrammatically the amount of mechanically held water found in some of the mortars after 28 days' storage in air. This was determined by drying the mortars at 110° C, then measuring their volumes. The loss of weight in drying was then calculated as percentage of the volume of the mortar.

If any unusual amount of water is retained by the lime under any conditions, it should be distinctly noticeable in these mortars. They were dried in air, thus affording the lime full opportunity to exhibit its water-retaining ability. The 28-day period was not too short nor too long for the experiment. At this age the lime should be at the height of its strength-producing activity—since

the strength of cement mortars and concretes does not reach its maximum until an age of from 90 days to one year.

The strengths of the mortars represented in Fig. 2 may be found by reference to Fig 1. For example, mortars represented on the lines AB and AC in Fig. 2 have compositions indicated by points on the lines AB and AC in Fig. 1. Strength is discussed elsewhere but it is seen from Fig. 2 that although replacement of sand by lime causes an increase in the amount of mechanical water present, replacement of sand by cement shows a greater increase. It is evident that these results do not support the theory of retention of water by lime in mortars.

The results of further experimental work on the behavior of the water content of the mortars are given in Table 2 and shown

TABLE 2.—RATE OF DRYING OF MORTARS IN AIR.

Percentage loss of water on basis of total water lost in air drying.

Time in days.	No. 61, 1:3 cement mortar.	No. 16, 1:3 mortar, 25% lime-replacing sand.	No. 56, 1:3 mortar, 25% cement-replacing sand.	No. 16 F, 1:3 mortar, 25% flint-replacing sand.
1	36.8	24.4	39.3	29.0
2	76.7	64.9	70.7	61.4
3	87.0	77.6	78.6	77.0
4	90.1	81.6	80.6	85.9
5	93.5	85.7	84.5	88.1
6	94.5	87.9	85.8	91.1
7	95.4	90.1	87.4	92.6
13	96.9	93.1	91.5	95.6
17	98.5	97.0	95.9	98.1
30	100.0	100.0	100.0	100.0
	On basis of air dry weight of mortar.			
Per cent water lost in air drying	12.21	12.50	9.40	11.75
	On basis of weight of dry materials in mortar when mixed.			
Per cent water used in mixing	17.50	18.83	17.50	17.06
	On basis of weight of dry materials in mortar when mixed.			
Per cent water used in hydration of cement	4.05	(5.63)	7.40	(4.76)
	On basis of weight of cement.			
Per cent water used in hydration of cement	16.2	(19.2)	14.8	(17.8)

diagrammatically in Fig. 3. Mortars Nos. 61, 16 and 56 of the main series and No. 16F, containing 25 per cent ground flint rock-replacing sand, were allowed to dry in air in the laboratory in a place protected from air currents and weighed at different times for a period of thirty days. The percentage loss of water for each period of time was calculated in terms of the total water loss in air drying. The flint rock was ground to pass a 200-mesh sieve.

As Fig. 3 shows, there was not a great deal of difference in the rate of drying of the mortars. A certain amount of credit must, of course, be given to the finer-sized materials whose use caused a small decrease in the rate of drying. Of these fine materials, hydrated lime, cement, and flint had approximately the same power of decreasing the rate of drying during a critical period of the life of the mortar (0 to 7 days after molding) when the structure of the mortar is being greatly affected by the colloidal developments of the cement.

As shown in Table 2 the percentage of water apparently used in hydration of the cement is greater in mortars Nos. 16 and 16F, containing lime and flint, respectively, than in No. 61, a 1 : 3 cement mortar. This indicates that the lime and flint either aided in hydration of the cement or that they themselves retained some water. The amount of water used in hydration of cement was calculated by the use of the following approximate formula:

$$W = \frac{B - A}{100 + A} \cdot 100$$

Where

W = percentage of water on basis of weight of dry materials in mortar when mixed.

A = percentage of water lost in air drying on basis of air dry weight of mortar.

B = percentage of water used in mixing on basis of weight of dry materials in mortar when mixed.

Discussions often appear in technical and trade journals stating that the addition of small amounts of hydrated lime to cement mortars or concretes increases strength. In such discussions

no reference is made to the effect of the addition of like amounts of cement to cement mortars.

The present series of mortars was not planned on the basis of addition of lime to the mortars. However, calculation will show that the addition of lime by weight of cement amounts to approximately the same as replacement of sand by lime, and the latter method is used in the present investigation.

In Fig. 5 is shown diagrammatically the effect upon strength caused by replacing sand by lime or by cement, in the case of water storage mortars. The curves were constructed partly by means of data given in Table 1 and partly by use of inter-

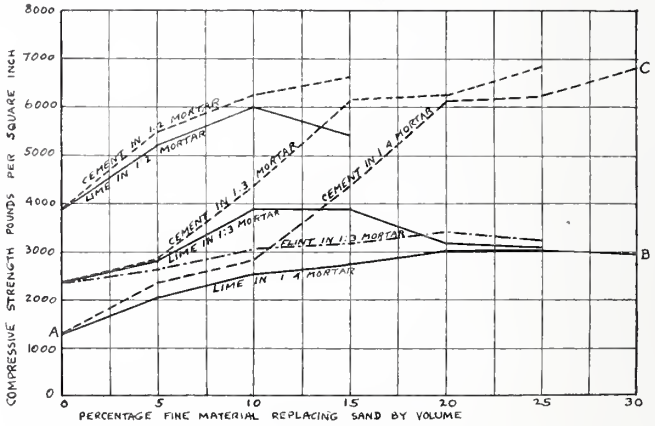


FIG. 5.—Effect of replacing sand by fine materials. Mortars stored 28 days in water. Proportions by volume.

polated values from Fig. 4. For example, the values for curves AB and AC in Fig. 5 are found at the proper points represented upon the lines AB and AC in Fig. 4. The line AB in Fig. 5 shows the increase in strength caused by replacement of sand by lime in a 1 : 4 mortar. The line AC shows the corresponding results when sand is replaced by cement, strength being higher in all cases. Similar results are found for the 1 : 3 and 1 : 2 mortars.

From these data the lime advocate will conclude that hydrated lime increases the strength of cement mortars; the cement advocate that cement increases the strength of cement mortars; the impartial observer that addition of hydrated lime increases

the strength of cement mortars but that the addition of like amounts of cement increases the strength much more.

In order to determine the effect of some fine material other than cement or lime, a number of mortars were made containing ground flint rock, passing a 200-mesh sieve. The compressive strength of these mortars was determined at the age of 28 days.

The results are given in Table 3 and those for water storage specimens are shown diagrammatically in Fig. 5. It is seen that the mortars containing hydrated lime were stronger than those

TABLE 3.—COMPRESSIVE STRENGTH OF MORTARS CONTAINING FINELY GROUND FLINT ROCK.

No.	Composition by bulk volume.				Compressive strength, pounds per sq. in. Air storage 28 days.			
					Flint.		Lime.	
	Sand.	Cement.	Flint or lime.	Water.	Air storage.	Water storage.	Air storage.	Water storage.
61	75	25	0	26.9	1225	2370	1225	2370
50	70	25	5	26.4	1645	2665	2110	2800
40	65	25	10	26.1	1925	3085	2540	3910
31	60	25	15	25.7	1935	3190	3110	3885
23	55	25	20	26.1	2265	3460	2925	3145
16	50	25	25	25.6	2215	3260	2580	3070

containing flint in all cases in air storage, and in all but two instances in water storage. The most probable reason for this behavior is that the hydrated lime is of smaller size of grain than the flint rock powder. The former is produced by slaking quicklime—giving an extremely fine powder. The latter is ground in ball mills about eight hours and could not be expected to be as fine as the hydrated lime.

Porosity.

Porosity is defined as the percentage of pore space in terms of the volume of the mortar. Porosity was determined as described under "Methods" and calculated by Purdy's formula:

$$P = 100 \frac{W - D}{W - S}$$

Where

P = porosity.
W = Wet weight of cured mortar after immersion in water.
S = Weight of wet, cured mortar suspended in water.
D = weight of cured mortar after drying at 110° C.

The relation between porosity and compressive strength is shown in Fig. 6. It is noted that the relation is quite accurate for the cement mortars but not at all accurate for the cement-lime mortars. It was attempted to obtain a closer relation by plotting strength against the product of porosity by cement-density

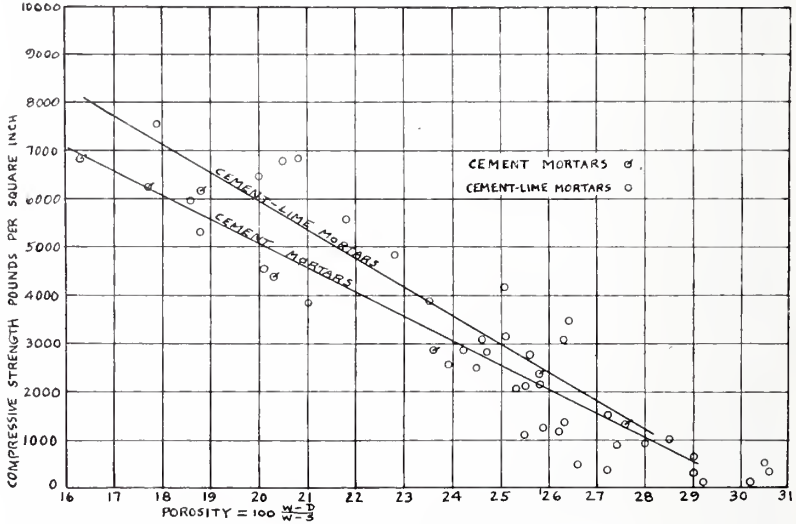


FIG. 6.—Relation between compressive strength and porosity at age of 28 days. Mortars stored in water.

(absolute volume of cement per unit volume of mortar). This method, however, proved unsuccessful. The most probable cause of these irregularities seems to be that complete saturation of the cement-lime mortars by water is not obtained by the usual method of making the test for porosity.

Water Ratio.

A function known as water ratio has recently received considerable attention in connection with the study of cement mortars

and concrete—due to the work of Abrams.[1] This function is found by dividing the volume of water by the bulk volume of cement in the mortar or concrete. The equation of the strength-water ratio curve was found to be of the form

$$S = \frac{A}{B^x}$$

Where

S = compressive strength in pounds per square inch.

x = water ratio $= \frac{W_1}{C_b} = \frac{\text{Volume of water}}{\text{Bulk volume of cement}}$.

A and B = constants whose values depend upon the quality of the cement, the age of the specimens, and the curing conditions.

To quote from Abrams, "This equation expresses the law of

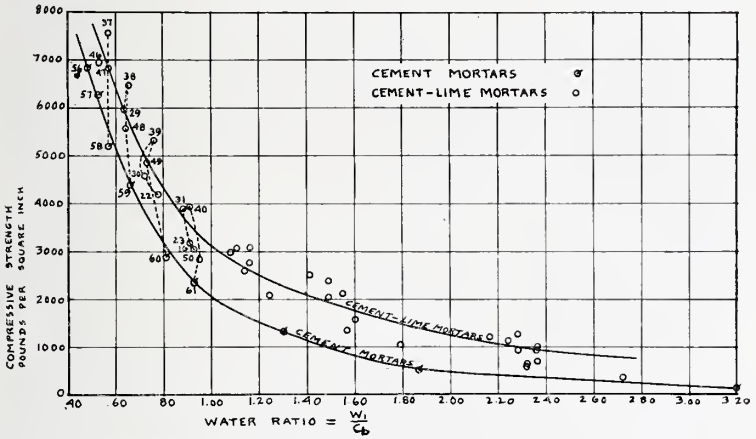


FIG. 7.—Relation between water ratio and compressive strength of mortars at age of 28 days. Stored in water.

strength of concrete so far as the proportions of materials are concerned. It is seen that for given concrete materials the strength depends on one factor only—the ratio of water to cement." The same statement applies also to mortar and neat cement.

In Fig. 7 the results for the present investigation are shown diagrammatically on the basis of water ratio. Neither of the

[1] *Engineering News-Record*, **80**, No. 18, 873 (1918).

curves shown conforms, throughout its entire course, to the equation $S = \frac{A}{B^x}$. Further mathematical treatment of these curves would serve no useful purpose since no other curves are available for mortars using the same materials and hence no comparison nor correlation could be made.

The fact that the curve for cement-lime mortars lies above that for cement mortars is not to be interpreted as meaning that the former are stronger in all cases. These curves were constructed for the purpose of subjecting the results to mathematical treatment and do not offer the proper basis of comparison, for practical purposes. The proper basis for such comparison has already been given in the discussion of the results shown in Fig. 5. For example, the builder wishes to erect a brick wall and for use in this wall has compounded a cement mortar. He wishes to increase the strength of this mortar without regard to other properties. He adds small amounts of either cement or hydrated lime (and water if necessary to obtain the proper consistency). He compares the strength of the two resulting mortars, ignoring the original mortar, since from the nature of the process it must be weaker than the two new mortars.

In Fig. 7, for example,, mortars Nos. 61 and 40 are not to be compared but Nos. 40 and 59. No. 61 is only the starting point for comparison. It is a 1 : 3 cement mortar. With 10 per cent lime-replacing sand, it becomes mortar No. 40. With 10 per cent cement-replacing sand it becomes mortar No. 59.

In the article of Abrams quoted above, the discussion is very brief and it is not clear as to what variables, if any, must be controlled in order that the plotted values for strength may fall on one curve. However this may be, the fact that the plotted points for cement mortars in Fig. 7 all fall upon a uniform curve indicates that other variables were sufficiently controlled in the present investigation.

For cement-lime mortars, the water ratio method of correlation is not as accurate as could be desired. For example, in Fig. 7 certain groups of mortars have practically the same water ratio and the same volume percentage of cement but widely different strengths. Such groups are indicated by the dotted lines joining the points involved. For example, in the group containing mortar

No. 59 the strength rises to a maximum with mortar No. 38, then decreases to that of mortar No. 29, with constant increase in amount of lime. Evidently the lime should be included in a ratio or function to be used for correlation with strength in order to afford a more accurate relation.

Density of the Mortar.

Density of the mortar is defined as the absolute volume of total solids per unit volume of mortar and is calculated by the use of the following formula:

$$D = \left(\frac{S}{s} + \frac{C}{c} + \frac{L}{l}\right)\frac{R}{V}$$

Where

D = density.

S, C, and L = weights of sand, cement, and lime, respectively, weighed out for mixing.

s, c, and l = apparent specific gravities of the sand, cement, and lime, respectively.

R = weight of material in the mold divided by the total weight of materials mixed, including water.

V = volume of mold.

In Fig. 8 is shown the relation between strength and density for certain groups of mortars. Mortars represented by points

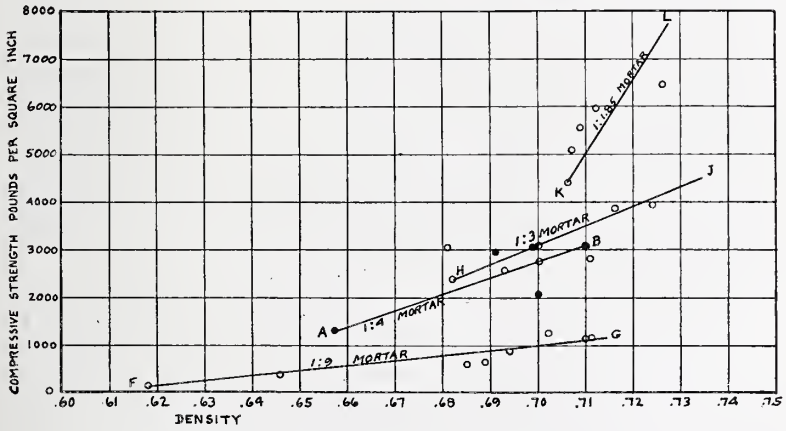


FIG. 8.—Relation between density and compressive strength of mortars stored in water 28 days.

along the line FG may be found represented upon the line FG in the triaxial diagram of Fig. 4; likewise, for the lines AB, HJ and KL. Each of these lines represents the effect of replacing sand by lime in a cement mortar. It is noted that for all the mortars, strength increases with increase of density. The point of highest strength on each of the lines corresponds to a point upon the line RR in Fig. 4 which is drawn through points of highest strength on lines parallel to FG. This means that hydrated lime acts as a void filler and increases density and strength. The maximum effect is observed at some definite amount of lime as shown by line RR in the triaxial diagram of Fig. 4.

It is desirable to represent the relation between strength and density as a continuous function. This has been done as shown in Fig. 9 where strength has been plotted against the cement-density product CD. In this product, C is the absolute volume of cement per unit volume of mortar and D is the density, as defined above. This seems to be the most accurate representation of the results. The curves of Fig. 8 show that the rate of increase of strength with density increases with the amount of cement and the next step is naturally to devise a function involving both density and amount of cement. The principles involved have been known for some time—having been stated by Feret[1] in connection with his studies of mortars.

It is noted that certain irregularities found, when the water ratio method of representation was used, do not appear when the results are plotted upon the basis of cement-density product. For example, points representing mortars Nos. 37 and 47, also Nos. 38 and 48, which lie directly above one another in Fig. 7, are well distributed along the curve in Fig. 9. This is due to the fact that the water ratio method does not take into account the density of the mortars.

In all three of the methods of representation employed (see Figs. 6, 7 and 9) the curve for cement-lime mortars lies above that for cement mortars. This fact is liable to be misinterpreted as meaning that all cement-lime mortars are stronger than cement mortars. No matter what theoretical or mathematical means is

[1] *Bull. soc. d'encour. l'ind. nat.*, 2, 1604 (1897).

used to determine the rate of change of strength of mortars, the means used to compare the relative value of cement and hydrated lime in the mortars will be practical and will not be upon the same

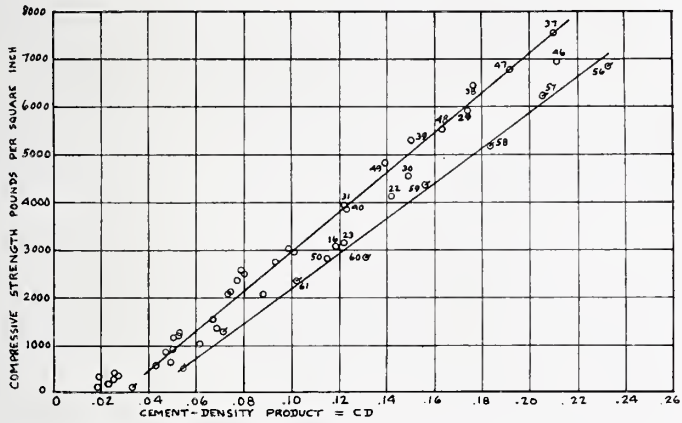


FIG. 9.—Relation between cement-density product and compressive strength of mortars stored in water at age of 28 days.

basis as the theoretical method. The practical basis for comparison has already been shown to be the percentage of cement or lime added to the mortar—the consistency being alike for all the mortars.

Conclusions.

In this investigation only one kind of lime, cement, or sand was used. Hence, the results cannot be given as much weight as would be given the averages of many tests using many different materials. They do give, however, an indication of the nature of the changes to be expected in strength of cement-lime mortars, due to change in proportions of the materials. It should be noted also that only one consistency was employed and the results would be different in degree if other consistencies were used. This would not, however, change the general relations since change in consistency would be accounted for by change in density.

Hydrated lime added to portland cement mortars in moderate amounts causes an increase of strength due to increase of density. Like amounts of cement added to the same mortars cause equal

or greater increases in strength. This is believed to be the proper and practical means of comparison which should be used in preference to any complex theoretical method. This is entirely aside from the other qualities produced in mortars by the use of hydrated lime. The present study has been of strength only.

Porosity and water ratio, which bear close relations to the strength of cement mortars, do not define accurately the relation for cement-lime mortars whose strength is governed by density and amount of cement as expressed by the cement-density product.

FUSIBILITY OF GRAPHITE ASH AND ITS INFLUENCE ON THE REFRACTORINESS OF BOND CLAY.[1]

By M. C. Booze.

The ash content of graphites used in the manufacture of graphite crucibles is considered to be detrimental both because it lowers the carbon content and because, upon ignition of the carbon, it exerts a fluxing action upon the body.

The various crucible graphites vary considerably in the per cent of ash which they contain. The Ceylon variety is probably the purest of the crucible grades, the lump containing from 2 to 15 per cent of ash and the chip from 5 to 30 per cent. The domestic graphites contain from 10 to 15 per cent of ash according to the process of milling and the specifications of the user. The average ash content of graphites used in the manufacture of crucibles is probably between 10 and 15 per cent. The ash is minutely associated with the graphite and of course can exert no fluxing action upon the body until the carbon is burned out. In a crucible, therefore, the fluxing action, if there be any, begins on the surface of the crucible, where oxidation first takes place, and proceeds inward only as fast as the ash is formed. The production of a dense skin on the crucible, which would accompany the softening of the clay and ash mixture, would retard the further penetration of gases and the combustion of the graphite so that the fluxing action of the ash would be materially retarded.

Laboratory Tests.

In the investigation, the softening points of graphite ashes and of ash and clay mixtures were determined in order to learn the extreme effect of the ash in lowering the refractoriness of graphite crucibles. The various graphites, which had been previously collected for experimental work on crucibles, were sampled by quartering down to about 200 grams. Each sample was then

[1] By permission of the Director, U. S. Bureau of Mines.

heated to 900° C in an electric furnace and held under a gentle current of air until all of the carbon had been burned out. Part of the ash so obtained was then mixed with dextrin and molded into cones of the same size and shape as the Orton standard cones. Other cones were made by mixing the ash with Mississippi bond clay in the ratio of 12 parts ash to 88 parts clay, or about the same proportion that the average crucible would show upon ignition of the graphite. The softening points of the cones were then determined against standard cones in a gas-fired pot furnace. The softening point of the clay used was determined in the same manner. The results are given in the table on the following page.

As a rule the ash from the Alabama graphite was more refractory than any of the others. The Ceylon and Canadian graphites gave ashes which were relatively quite fusible. The ash from the Pennsylvania graphite also had a low softening point.

The mixtures of clay and ash did not show the variation in softening points that might be expected from the results with the ashes alone. With the exception of three of the mixtures, a variation of only three cones was noted. The mixtures containing the ash from Alabama graphite show the highest softening points while the mixture containing ash from Canadian graphite gave the lowest softening point. The ash from Ceylon graphite, while showing a low softening point itself, gave a high softening point when mixed with clay. There appears to be little relation between the softening point of the ash and the softening point of the clay and ash mixtures.

None of the mixtures and only three of the ashes show softening points low enough to be effective at brass melting temperatures. Fourteen of the graphites gave ashes which would soften at steel melting temperatures while eight did not. Nine of the clay-ash mixtures softened at points corresponding to the highest temperatures used in crucible steel melting, three were low enough to be effective at the average temperatures used, and ten were above maximum temperature—assuming that cone 26 corresponds to a temperature of 1600° C, as given in the latest determinations.

Materials used.	Softening point of ash (cones).	Appearance of ash.	Softening point of clay-ash mixtures (cones).	Appearance of clay-ash mixtures.
No. 1 Alabama graphite	17	Black	26	Slightly swollen
No. 1 Alabama graphite	18	Black, slightly swollen	27	Gray, badly swollen
No. 2 Alabama graphite	32	Black	29+	Gray, slightly swollen
No. 1 Alabama graphite	30	Black	29+	Gray, slightly swollen
No. 2 Alabama graphite	20	Black	27	Gray, spotted
Alabama graphite dust	30	Black	29+	Gray, swollen
Alabama graphite dust	31	Brown	29	Gray, swollen
Alabama graphite dust	31	Brown	29+	Gray, swollen
No. 2 Alabama graphite	32	Brown	28	Buff, swollen
No. 2 Alabama graphite	17	Brown	26	Gray, slightly swollen
No. 1 Alabama graphite	32	Brown	29	Gray, swollen
No. 1 Alabama graphite	18	Brown	26	Gray, slightly swollen
No. 1 Alabama graphite	23	Brown	27 1/2	Buff, swollen
No. 1 Alabama graphite	17	Brown	26	Gray, slightly swollen
No. 1 Alabama graphite	32	Brown	26	Gray, slightly swollen
No. 1 Alabama graphite	16	Black	26	Gray, slightly swollen
No. 2 New York graphite	8	Black	23	Iron spots, slightly swollen
New York graphite dust	3	Black	26	Gray, swollen
Canadian graphite, crucible grade	1	Black	17	Gray, swollen
Ceylon graphite, crucible grade	2	Black	26	Iron spots, slightly swollen
No. 1 Penna. graphite	20	Black	19	Black, swollen
Pot shell, crucible grade	9	Black, swollen	26	Gray, swollen
Softening point of Mississippi clay		Cone 30		

A degree of softening below that obtained in the clay-ash cones would undoubtedly be detrimental to a crucible, but since the clay and ash in a crucible are not so intimately blended as in the cones, the softening point of the crucible mixture may even be higher than herein registered. The clay used for this work showed a softening point of cone 30—which is somewhat low for steel crucibles. Clays of the type used for steel crucibles do not vary widely in their composition and the use of a more refractory bond clay should still further raise the softening points of the mixtures.

Conclusions.

The softening point of a graphite ash is not a true criterion of its action in a crucible body.

The tendency of graphite ash to lower the refractoriness of a crucible is not evident at brass-melting temperatures and would seldom be detrimental even in steel melting crucibles.

The ash from Alabama graphite is less effective as a flux than the ashes from the Ceylon, Canadian, Pennsylvania or New York graphites.

Bureau of Mines,
Mining Experiment Station,
Columbus, Ohio.

CLAYS FOR USE IN PAPER-MAKING.

By Ralph B. Roe.

For a great many years it has been customary to introduce a certain amount of mineral fillers or loading materials into the suspension of fibers in water which forms the basis of paper. In the conversion of this suspension of fibers into a sheet of paper, a considerable proportion of the mineral filler is lost, while the balance remains associated with the fiber in the paper sheet—contributing in no small degree to its physical characteristics.

Without knowing the effects of such an addition of loading materials, one might be inclined to consider the practice more or less reprehensible for the same reason that the introduction of fillers, loading materials, and "make weights" constitutes an adulteration in the case of many well-known articles of commerce and every-day use. Although the introduction of relatively cheap and heavy fillers actually does decrease the intrinsic value of the paper, especially since the latter is almost always sold on a basis of weight, the physical qualities imparted to the sheet really constitute the primary object in view, and it is no exaggeration to say that without fillers it would be quite impossible to manufacture a product that would give satisfactory service in some of the widest and most essential applications in which paper is used.

A sheet of machine-made paper is essentially an interlaced structure of individual fibers and as such will show not only an uneven surface but it will also be apparent that owing to its structure there will be innumerable interstices between the individual fibers comprising the sheet. When it is considered that for printing purposes a physically even and close texture is a prime requisite, it becomes evident that a sheet of paper with these qualities is not perfectly adapted for fine printing work. The introduction of a filler acts toward filling the interstices or voids in the paper. At the same time the sheet becomes softer and more pliable and, the surface being smoother, is capable of receiving a higher mechanical finish in the course of manufacture.

Even more important is the tremendous effect on opacity. A thin sheet of paper of a given fibrous composition and thickness would be utterly useless for printing purposes compared to the same sheet heavily loaded with a filler. The well-known thin printing papers, referred to as bible and India papers, owe their amazing opacity to the relatively high content of filler. It should be understood, however, that the desirable effects of loading are gained at the expense of strength, and it is in only those paper stocks that are so manipulated as to yield extraordinary strength that high percentages of filler can be employed without seriously affecting the wearing qualities of the paper.

In addition to incorporating mineral agents into the sheet, there is another and somewhat different field of application. This consists in applying the mineral ingredient mixed with a suitable binder to the surface of the sheet. In this way a surface is obtained which is susceptible of receiving a very high finish. This product, known as coated paper, is used in high-grade printing and illustration work.

The number of mineral ingredients used for filling and coating papers is comparatively restricted. For filling purposes may be mentioned clay, sulphate of lime (known in commerce under various names such as pearl hardening, crown filler, terra alba, gypsum, etc.), talc, asbestine or agalite, and blanc fixe, or precipitated sulphate of barium. For coating, clay, blanc fixe, and satin white are the most important. Satin white may be described essentially as a mixture of precipitated sulphate of lime and hydrate of alumina with more or less hydrate of lime.

Of all the materials in use for loading and coating purposes, clay is by far the most important—a fact which is largely due to the relatively low cost of this material. The source of the clay used in the finest grades of paper has been for many years almost exclusively confined to England. An excellent natural product, methods of production yielding a very high-grade material, and favorable shipping conditions, all operated to cause the continued use of English clay and conversely inhibited the growth and development of domestic clays for this purpose. For a number of years American clays have been exploited for paper-making purposes but with a few exceptions they were of decidedly poor

quality. Owing to war conditions, however, there has been considerable difficulty in obtaining English clay and increased attention has been directed to clay of domestic origin. Before making any comments on the relative merits of English and American clays, some mention should be made of those characteristics by which the quality of a clay is rated. The most important points to be considered are: (1) Color; (2) Grit; (3) Retention.

The value of any clay for either filling or coating purposes is largely affected by the color. There is a decided tendency toward the yellow in natural colored paper and it is the universal practice to correct this yellow cast by the addition of a small amount of a blue pigment or dye. Since the amount of clay contained in the more common grades of printing papers will run as high as 20 per cent, it is obvious that the color of the clay determines in no small degree the color of the sheet. While a high white is an important consideration in clays used for fillers, it is especially to be desired in clays used for coating. In the latter case all of the clay lies in a layer or coating on the surface of the sheet and the color of the paper is entirely determined by the color of the clay.

The color of clay, insofar as it concerns paper-making practice, is judged entirely by arbitrary standards. For coating and high-grade printing papers the highest possible white is desired, whereas for cheap printing papers, a clay with a decided gray or yellow cast may be perfectly satisfactory.

To compare the color of any standard sample and an unknown specimen, a small amount of each is reduced to a fine powder and the two are then dried for a short time to ensure approximately the same moisture content. A portion of each is placed on a glass plate or sheet of white paper in such a way that the two samples lie side by side. With a paper or spatula the two samples are flattened out carefully so that they present a smooth, even surface with a common line of demarcation between the samples. The slightest difference in color shows up plainly by contrast.

The second desirable quality in a clay concerns its content of grit. By grit is included those particles of a sandy or siliceous nature together with those occurring in flakes or leaflets and micaceous in appearance. Siliceous grit is particularly objection-

able for the reason that it causes an excessive amount of wear on the fine wire of the paper machine. It moreover is likely to cause breaks in the paper during the progress of the sheet from the wet-end to the dry-end of the paper machine, and finally it manifests itself unmistakably in the finished sheet, by pin-holes, specks, etc. Micaceous grit is objectionable, largely because of its showing up as shining specks in the paper. These specks are very likely to cause trouble in printing—due to the particles picking off.

The estimation of grit is easily and quickly made by weighing a definite quantity of the sample (10 grams or more), transferring into a beaker or cup, and adding water. As soon as the clay has disintegrated, the contents of the beaker are transferred to a 200-mesh screen and the residue on the screen worked carefully with the finger while a small stream of water plays on the sample until nothing remains that may be washed through the screen. The residue is then dried, transferred to a paper, and weighed. It is expressed as a percentage of the original sample. The best clays do not contain more than 0.20 per cent of grit and there are many samples showing less than 0.10 per cent.

The retention of a clay is a term used to express the percentage ratio between the amount of filler used and the amount retained in the paper. The amount of filler retained is the sum of a number of physical conditions including certain characteristics in the clay itself together with the nature and treatment of the fibrous material composing the paper. Since the clay not retained is usually lost, the percentage retained has an appreciable bearing on the cost of production.

There is no satisfactory method for directly estimating the retention of a clay, and the necessary data is usually accumulated by actual test in the paper mill. Although there are no direct methods for estimating retention, certain indirect methods have been suggested which furnish comparative data on the retention of clays. The principle involved is graduated levigation or elutriation under carefully controlled conditions.

Attention was directed earlier in this paper to the recent tendency to develop American clays for use in paper-making. For many years there was a distinct prejudice in most quarters against local clays, it being claimed that the English article is of better

color, contains less grit, and possesses to a higher degree those qualities which are peculiarly essential in a coating clay. As far as color is concerned, domestic clays tend to a yellow tone whereas the imported article is more of a blue-white. It is a fact, however, that some of the higher grades of English clay are artificially colored with a blue coal-tar dye, which undoubtedly accounts for the superior color. In some cases ultramarine has been used for the purpose. This pigment is unstable under conditions which might easily obtain in paper-making, in which case the color imparted would fade. While the average English clay is better in color than the average American clay, some of the domestic clays now on the market are of good color and are admitted in most quarters to be quite satisfactory for filling purposes.

The prejudice against domestic clays is to a large extent founded on the excessive amount of grit which was shown in the earlier grades. The evil effect of grit in clay is easily demonstrated and any feeling engendered against a clay on this ground is quite justified. In the past few years, however, some of the domestic clays have been so far improved in this respect that they do not suffer by comparison with the imported product. It must be admitted that the reputation acquired in the past for excessive grit has been difficult to overcome.

For coating purposes, the view seems to be quite universally held that domestic clay does not fulfill the requirements of the trade. Excepting color, which must be high, there are certain rather obscure characteristics which render the English clay peculiarly well fitted for the purpose. It is probable that these desirable qualities are intimately connected with the plasticity. Although considerable research work has been carried out on the subject of paper-making clays, the matter would seem to offer great possibilities for further work with especial reference to the coating problem.

In conclusion, it is not unduly optimistic to hope that even as domestic clays have been increasingly used for fillers, continued and thorough research will prove that the more exacting requirements of the coated paper manufacturer are not inherently impossible of being met by our American clays.

ARTHUR D. LITTLE, INC.,
CAMBRIDGE, MASS.,
January 30, 1919.

AMERICAN CERAMIC SOCIETY.

Acquisition of New Members during January, 1919.

Associate.

Allcock, F. T., American Encaustic Tiling Co., Zanesville, Ohio.
Anderson, F. E., 905 Commonwealth Bldg., Pittsburgh, Pa.
Becket Frederick M., Union Carbide Co., Niagara Falls, N. Y.
Blachford, H. L., The Fairite Co., 669 W. Ohio St., Chicago, Ill.
Brinkman, S. G., Fords, N. J.
Brown, Lawrence H., 1835 Indianola Ave., Columbus, Ohio.
Burchfiel, B. M., Mellon Institute, Pittsburgh, Pa.
Callahan, H. D., 434 E. Long St., Columbus, Ohio.
Carling, J. P., China Products Co., Zanesville, Ohio.
Chang, C. Y., 228 W. Lane Ave., Columbus, Ohio.
Davis, Harry E., 396 Stoddart Ave., Columbus, Ohio.
Douda, Henry W., 11 E. 18th Ave., Columbus, Ohio.
Evans, A. W., Zanesville, Ohio.
Faulkner, Karl B., Corning Glass Works, Corning, N. Y.
Hepplewhite, J. Wilson, 1835 Indianola Ave., Columbus, Ohio.
Koos, E. Kenneth, 1210 Dennison Ave., Columbus, Ohio.
Kraner, Hobart M., 61 Welch Ave., Columbus, Ohio.
Kreger, John M., Monument Pottery Co., Trenton, N. J.
Laird, Charles, 16 Fifteenth Ave., Columbus, Ohio.
Liang, Ching-Hsia, 174 W. Lane Ave., Columbus, Ohio.
Long, Russell N., 1932 Waldeck Ave., Columbus, Ohio.
Oakley, Walter W., 32 E. 2nd St., Corning, N. Y.
Odelberg, A. W., Gustafsberg, Stockholm, Sweden.
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EDITORIALS.

THE CONVENTION.

The twenty-first annual meeting of the American Ceramic Society, Pittsburgh, Pa., adjourned *sine die* at six o'clock on the evening of the sixth of February, and it was a wonderful and genuine success. There had been anxious thoughts in official circles as the time drew near for the meeting. The year had been full of activities, but some of the developments were still in the experimental stage. There had been a large influx of members but the fear of undue dilution which had existed in the old days had not been entirely banished. Could the spirit of the Society be kept as keenly alive among a thousand men as it had been among two or three hundred? Would the passion for research not be reduced in intensity by being spread too thin? These and other questions had yet to be answered.

At the Fort Pitt Hotel on Sunday afternoon the answers began to come. The well-known faces showed themselves, keen, alert, and friendly as before. The atmosphere grew warmer and more pleasant as the hours passed until all doubts vanished and unknown quantities produced their own solution.

The Board of Trustees met on Sunday evening and remained in session until the early hours of Monday morning. It was then decided that all actions of the Board should be published from time to time in the Journal, so that it is not necessary to detail them here. Let it suffice to say that the Board is unanimous in the continuance of a broad policy of development and advance without sacrificing the principles of unity and altruism for which the Society has always stood.

The business meeting on Monday was held in the Assembly Room and although this room was capable of seating a hundred and fifty persons, it was almost inconveniently crowded. The Secretary had provided about 250 badges, which in ordinary times would have been more than enough, but even on the first day the stock ran low and before noon on the second day it was entirely exhausted.

The meeting was called to order at 10 A.M. by President Staley, who delivered the annual address in which several important suggestions were offered relative to the future activities of the Society. The address was referred to a committee of three, who duly considered the points raised and referred them to the proper committees.

The routine business was then disposed of in the usual way. The report of the Board of Trustees was read and the finances were shown to be in a flourishing condition. Five thousand dollars had been invested in Government securities and there was a cash balance of eighteen hundred dollars. The membership roll contained 1036 names and more were being added as fast as the necessary action could be taken.

The reports of committees consumed some time and a few of these were postponed to the final business session. Mr. A. F. Greaves-Walker, Chairman of the War Service Committee, told of the ways in which members of the Society had been able to assist the country to overcome the difficulties occasioned by war conditions. Dr. E. W. Washburn reported that the National Research Council was to be reconstituted on a peace basis and said that the Society was invited to nominate one of its members to the Council. Mr. Purdy, speaking for the Membership Committee, announced that their goal was 2,000 members by the end of the year. Mr. Beecher, Chairman of the Committee on Standards, gave a résumé of their report and stated that the full report would be printed and issued to the members for criticism or confirmation. The Committee on Rules was charged with the duty of considering several suggested changes which were to be reported to the Society before final adjournment so that action might be taken by ballot.

The formation of Professional Divisions was discussed and the opinion was generally expressed that these might be expected to constitute a source of great strength to the work of the Society.

Dr. E. W. Washburn asked that a committee be appointed to define the meaning of the term "ceramic" and to take steps to place a definition, accepted by the Society, before the publishers of dictionaries and encyclopedias.

An adjournment for luncheon was taken at one o'clock and soon after two o'clock the second session was called to order.

The reading of papers now began, the first group being of contributions relating to refractories and especially to graphite crucibles. These papers will be printed in full in due course and it will suffice to say that the subjects were treated with technical thoroughness.

Monday evening was set apart for the organization of Professional Divisions. The Society met as a whole in the Assembly Room at eight o'clock and Mr. Purdy spoke of the importance of the work which the Divisions might be expected to accomplish. After some discussion as to methods of procedure, the members separated into groups for the purpose of organization. Divisions were suggested on enamels, glass refractories, abrasives, porcelain, brick and tile, stoneware, terra cotta and faience, wall and floor tile, and white wares. Not all of these were formed at once, but steps were taken which led or will lead to complete organization. Each Division is to have its own officers. A general supervision is to be exercised by the Board of Trustees but, having regard only to the rules governing the Society in general, Divisions will be expected to work out their own plans.

Late in the evening the Board of Trustees held a meeting at which the qualifications of a number of associates nominated for elevation to membership were reviewed.

On Tuesday morning the session met in the banquet hall. By request of the Division of Enamels, the papers relating to this subject were called first and following these were the papers on glass and glass pots. At two o'clock a joint session was held with the United States Potters Association, of which a large delegation was in attendance from East Liverpool and the neighbor-

ing pottery centers. On this occasion there were probably four hundred persons present, thus making the meeting by far the largest ever held by the Society. Papers on white ware manufacture were read and discussed, and when the closing hour arrived the pottery men adjourned to the Assembly Room, where the White Ware Division was organized with Mr. C. L. Sebring as chairman. At the same time the Division on Glass met in a committee room and discussed the special problems of their subject.

The dinner on Tuesday evening was a notable event. The Society has not as a rule been favorable to the banquet idea. This was the twentieth anniversary, however, and the occasion seemed to call for a celebration. An attempt was made to secure H. A. Wheeler, the first president, as chairman, but he was in far-off Texas and could not be reached. The Board of Trustees, therefore, delegated Professor C. F. Binns, the senior president present, to take the chair. Notwithstanding the loss of the menus and song books, the pianist rose to the occasion and with due assistance from Messrs. Purdy and Forester the songs were rendered. The toast list was arranged in two groups of three subjects each: "What We Are," "What We Were," and "What We Will Be," by President Staley, Professor Bleininger and Dr. Tillotson, respectively, and "Scientifically Speaking," "Practically Speaking," and "Periodically Speaking," by Dr. Washburn, Mr. E. W. Wells and Mr. L. E. Barringer. Mr. Wells kindly consented to speak at a few minutes' notice as the expected speaker was not present.

Two hundred and seventeen persons were present, including a small number of ladies. Perhaps if this becomes an annual event the fair sex may be more numerously represented.

On Wednesday morning the Society was compelled to return to the Assembly Room, the larger room being otherwise occupied. The result was a rather crowded condition but everyone made the best of it. The reading of papers was continued and the end of the program was reached by four o'clock in the afternoon. The vice-president then took the chair and the adjourned business meeting was resumed. Several reports which had been postponed were read. The chairman announced that Mr. Samuel

Geijsbeek, the man who had 21 years before suggested the formation of the Society, was in the room and was requested to show himself. Mr. Geijsbeek accordingly came to the table and was received with an ovation.

The result of the annual election was then announced: R. T. Stull, President; R. H. Minton, Vice-President; C. F. Binns, Secretary; R. K. Hursh, Treasurer; and M. F. Beecher, Trustee. These gentlemen were then duly installed and the meeting stood adjourned.

The outstanding characteristics of the Convention were the spirit of progress and genuine interest in the future of the Society.

CONVENTION TRIPS.

Thursday, February 6th, was devoted to excursions to places of special interest to members of the Society. One party set out to visit the glass plants at Rochester and Sharpsburgh. A second party visited the Bureau of Standards, the Bureau of Mines, and Mellon Institute. A third party proceeded to the plant of the Harbison-Walker Refractories Company.

At the Bureau of Standards the history of the Arsenal, built in 1814, was related and the modern laboratories were inspected. The recently erected plant for the manufacture of optical glass proved of great interest as did also the processes of the manufacture of the pots of fire clay and porcelain. The giant testing machines and the freezing tests of structural materials were explained.

After lunching in East Liberty, the party went to the extensive laboratories of the Bureau of Mines, where inspection was made of the Government equipment for investigation and research. A test of gas masks under actual mine conditions proved of great interest; Messrs. D. A. Lyons and R. T. Stull guided the party through the Bureau. At Mellon Institute Dr. E. W. Tillotson explained the work which was being done and an inspection of the laboratories followed.

At the plant of the Harbison-Walker Refractories Company, Mr. Town, the chemist in charge, conducted the visitors through the laboratory and exhibited the special furnaces used for attaining extremely high temperatures. The manufacture of silica brick was new to many and very interesting. The remarkable effect of a small percentage of lime on the crushed rock was noted, as was also the fact that in burning the brick a large allowance had to be made for expansion. It was also noted that on account of underground moisture, the waste heat was carried to the driers by overhead ducts. Many thanks are due to the staff of the Harbison-Walker Company for the hospitality shown to the visitors.

In the afternoon a group of members proceeded to Sharpsburg on the Allegheny River and there visited the bottle factory of the H. J. Heinz Company. Mr. Ripley, of this company, conducted the party through the plant. The Owens bottle machines and glass melting furnaces used in connection therewith proved of great interest.

The trips, although informally arranged, proved thoroughly enjoyable and profitable. It is to be hoped that at the future conventions particular attention will be given to this phase of the meeting. It is true that the Summer meetings are devoted for the most part to inspection and sight-seeing trips, but as our annual meetings are held in different parts of the country each year every opportunity should be given to those in attendance to visit the ceramic plants of interest in the vicinity of the meeting place.

ORIGINAL PAPERS AND DISCUSSIONS.

REFRACTORIES FOR THE ZINC INDUSTRY.

By M. Grover Babcock.

Introduction.

One special branch of the ceramic industry which has not been discussed in the publications of the American Ceramic Society is the subject of zinc refractories, which consists principally of the manufacture of retort and condenser shapes as the finished products and plastic fire clays, grog and coke as the raw materials. The bond clays, however, are by far the most important part of the retort batch and are of interest to the clay worker. It would seem desirable, therefore, to bring to the attention of the Society a résumé of the requirements of retorts; the materials of a retort batch; their chemical and physical properties; tests of refractory bond clays used in retorts; preparation and the proportions of the materials used for the batch; treatment of the batch; manufacture of the product; and the life of the retort and condenser.

The principal shapes under the general term zinc refractories are, in the order of their importance, retorts (spelter vessels), condensers (spelter catchers), and high-grade fire brick. The first two are least familiar to most ceramists. Retorts are usually either cylindrical or elliptical in shape, 48″–51″ long, 8″ in diameter, inside, and 1″ thick, weighing about 134 pounds when burned. The condensers are 14″ × $5^1/_2$″ × $2^1/_4$″, inside measurements, and are leg-shape or conical in form, being bulged out near one end, with a belly to collect the spelter.

The requirements of clay retorts used in the distillation of zinc ores are very exacting and may be summarized as follows:

First: They must possess the property of sufficient refractoriness to withstand the high heat of the melting furnace and to maintain a maximum heat conductivity to support the endothermic reaction of the ore during the reduction.

Second: A great plasticity of the bonding clay is desired, to cement together the grains of calcined grog, or chamotte, into a compact mass.

Third: Retorts must possess considerable mechanical strength and toughness, especially in the dry state, as they are subjected to several handlings before being placed in the furnaces. Strength is also necessary to maintain the shape while under load of ore and coke at smelting temperatures, 1300–1400° C.

Fourth: They should become dense at 1300–1400° C or lower, thereby producing a structure with a minimum porosity, which makes them impervious to the zinc vapors at these temperatures and chemically inert to the corroding action of the various slags produced by impurities in the gangue of the zinc ores.

Fifth: The retorts must be made with thin walls to obtain the maximum conductivity necessary for the endothermic reaction taking place inside the retort and at the same time maintain the heat flow necessary for the continuation of that reaction without a drop in temperature.

Sixth: The properties of retaining a sound structure after molding and of drying free from incipient cracks, and of burning to a dense product, are especially essential to a good retort.

These requirements are dependent primarily on the raw materials used. Of the raw materials, the bond clays are of primary importance and the grog secondary. Unfortunately, the ideal bond clay for this type of refractory has never been discovered. The clays which have been found most satisfactory by years of practical experience are those from the Cheltenham district in Missouri and those from the Woodbridge district in New Jersey. These clays possess the properties demanded to a certain degree and are comparatively cheap, but there is still room for improvement. If the perfect materials for retorts are discovered, the zinc industry will not be slow in making use of them.

In considering a clay for zinc refractories, a few general chemical properties which are important can be enumerated. It should be low in ferric oxide, lime and alkalies, which tend to increase its fusibility and decrease its refractoriness. These clays should contain no free silica, provided the ore to be smelted is a basic gangue. However, with a silicious ore, a clay containing a little free silica is an advantage. The character of the ore governs the kind of clays to be used in the retorts in a small way, but it is not so important as many of the other physical properties which a good plastic refractory clay must possess to be suitable for retort-making. From a physical standpoint, the properties of a perfect retort should be maximum heat conductivity, refractoriness, mechanical strength, toughness, minimum porosity and low coefficient of expansion. In a large measure, these properties are dependent upon the raw materials, the size and amount of grog in the batch, the preparation and ageing of the batch, and the methods of manufacture.

Raw Materials.

Bond Clays.—Plastic clay used as the bonding agent in the retort batch should possess a considerable water shrinkage to insure a complete binding together of the grog particles. The refractoriness of clays of this nature ranges between Seger cones 30–31, although a clay of sandy nature which becomes dense at a lower temperature with the property of standing up over a long heat range before showing vesicular structure would not be condemned on account of its low softening point. In fact, many of the clays actually used in the retort industry are only tested to stand above 1630° C. The linear shrinkage of clays used for retorts ranges from about 4.0 to 6.5 per cent after drying, first at room temperature and then at 110° C. The porosity-temperature requirements for these clays would range from 10.0 per cent at 1150° C to approximately 5.0 per cent at 1250° C and to maintain this minimum porosity up to 1400° C before becoming vesicular.

Clays which have been used quite extensively for retort and condenser mixes are found in two localities in the United States—one a dense burning sandy clay from the Woodbridge district, New

Jersey; the other a plastic fire clay of great shrinkage from the Cheltenham district in Missouri. Here one can combine the good qualities of both by mixing the two in the proper proportion. The resultant mix has a softening point slightly lower than either of the original clays, the softening point of a mixture of these clays being lower than the arithmetical mean of the softening points of the two components.

The clays after being purchased from the miners are oftentimes stored in piles in the open, where they go through a weathering and a partial disintegration before they are used. It has been stated[1] that the extra cost of handling and the loss of clay incurred by this method do not justify the advantages gained by the weathering, and in some plants this procedure is not in favor.

The Grog or Chamotte.—Grog, or chamotte as it is sometimes called, may come from several sources. Probably the best grog is the calcined flint clay from the Clearfield district of Pennsylvania. The clay is ground, pugged with water, and ejected from a pug mill in square blocks, which are then dried and burned in an ordinary kiln. These burned blocks are reground and screened according to size, and finally stored for use. The longer life of retorts when the flint clay has been the source of grog leads one to favor this source of grog as the most satisfactory and economical in the long run, notwithstanding the fact that, due to its double preparation, the first cost appears large.

Other sources of grog are old retorts, brick bats, pottery saggers, and salvaged glass pots and tank blocks. In using old retorts for the grog source, one has a chamotte which has undergone a complete change in the furnace through the action of the zinc vapor absorbed in it, which with the alumina of the plastic clay has formed a mixture of zinc spinel and tridymite. This is evidenced by a blue coloration of the clay found in old retorts; this color is sometimes a deep purple. Thus, old material from retorts is of limited refractoriness and mechanical strength. Retorts made from this grog used in large amounts tend to bend and break at high temperatures, so that if the old materials must be used, the proportion of them in the batch should be very low.

[1] C. P. Fiske, *Bulletin A. I. M. E.*, **130**, 1719 (1917).

Of the grog obtained from other sources, such as waste products, that found in old pottery saggers offers the best possibilities, as it has been least contaminated by impurities that would tend to decrease the strength of retorts. Grog from other sources, except new flint clay, tends to have the disadvantage of imparting low mechanical strength to the retorts, and hence, as a result, a larger breakage loss in handling and a lower service record in the furnaces should be expected.

Coke.—Additions of coke dust and pulverized coke up to 10.0 per cent of the batch are now commonly made. This imparts a smoothness to the walls of the retort and gives a greater density and better working properties in the presses. The retort, where coke has been used, has the appearance of a graphite crucible. It has been said that the coke tends to prevent absorption of zinc vapor and diminishes the corrosive action of clays and metallic oxides of the gangue. However, opinions vary regarding the advantages of coke; but, whenever it is present, the shrinkage is reduced and a larger proportion of fine grog can be used in the batch without the danger of excessive cracking. Coke has also been found to increase the density of the vessel. It is the opinion of the writer that additions of domestic graphite may be used to good advantage in the place of coke, provided, of course, that the cost is not prohibitive.

Silica.—Sand has also been incorporated in the batch used in the zinc industries of Belgium and in other sections of the continental zinc industries where a silicious ore is smelted. Where additions of silica to the batch are made, the retorts cost less than those rich in refractory clay and they can be molded with thinner walls, thereby producing a gain in the rate of heat conductivity.

Glazes.—Some retort-makers glaze their retorts with a frit of fluorspar and zinc sulphate. The advantage which is claimed is a denser retort whose inner surface is more impervious to the zinc vapors, cutting down the loss due to filtration and absorption. The action of the frit takes place in the annealing furnace. After the fluorine has been expelled, the retorts assume a very dense inner surface. Mixtures of soda, flint and fluorspar are also used to accomplish this result.

Tests for Retort Clays.

The most reliable test for ascertaining the suitability of refractory clays for zinc retorts is that of the actual service record in the furnace of a large set of retorts. However, this is a lengthy experiment, as considerable time must elapse between the first step in the production of an experimental batch and its final trial in the furnaces, and, again, there are so many outside factors that influence the results that it is often difficult to decide just the exact value of the clay under test. Common factory practice has been to adopt a definite batch once the proper clay and chamotte have been determined for the particular ore smelted. Complete chemical analyses have been found to give little information that would guide the retort-maker in the selection of clays for the refractories. However, there are some physical tests utilized in the laboratory and factory which, the writer has been informed, work very satisfactorily.

At the Palmerton pottery of the New Jersey Zinc Company the following tests are employed to good advantage:[1]

Fusion test, to see whether a clay softens below 1630° C.

Dry shrinkage, linear, at 1230° C.

Burned shrinkage, linear, at 1230° C.

Porosity at 1230° C, as determined by the absorption method over a period of 12 hours.

Tensile strength of clay, 1 weeks' rotting;

Tensile strength of clay, 2 weeks' rotting;

Tensile strength of clay, 3 weeks' rotting;

Tensile strength of clay, 4 weeks' rotting.

In carrying out these tests, the clays are mixed with from 16.6–18.7 per cent of water, to a consistency approximating that used in the pugging machines. Figure-eight molds are used to form the test briquettes; the clay is tamped or thrown forcibly into the mold, and, on removal from the mold, the clay briquettes are allowed to dry at least one day at room temperature and one day at 110° C. One-half of the briquettes are tested for tensile strength and the others are reserved for porosity and burned tensile-strength tests. Along with every test on a new clay or

[1] C. P. Fiske, *Op. cit.*, p. 1721.

grog, a set of briquettes of the standard clay and grog batch is always carried through the test, for the purpose of obtaining comparative data. In all these tests it is the aim to obtain material that gives a superior mix to the standard in present use or to obtain equally as good results at less expense. This being the case, no definite limits have been established except that the clay must not soften below a temperature of 1630° C.

According to Fiske's conclusion,[1] the tensile-strength test is the most important, for it is indicative of the toughness of the vessel, and therefore of its ability to stand handling both before and after burning, as well as to withstand the strains and bumps received during the process of cleaning-out the retort. Tests of the nature of those mentioned are not conclusive, but by standardization and comparison they can be made very useful and will often prevent expensive failures which might otherwise occur in a trial of new materials.

The writer feels that these tests are very good in so far as they go, but submits that a more complete schedule of tests would, in the long run, give more standardized results and complete definite information on each and every clay tested. The tests to which the writer has reference are the following:

(1) Actual softening point of the clays and retort mixes in Seger cones.

(2) Slaking test.

(3) Transverse test on samples dried at 110° C.

(4) Transverse test on samples burned at 850° and 1150–1425° C.

(5) Tensile strength test on sample dried at 110° C, no rotting;

Tensile strength test on samples dried at 110° C, 1 week's rotting;

Tensile strength test on samples dried at 110° C, 2 weeks' rotting;

Tensile strength test on samples dried at 110° C, 3 weeks' rotting;

Tensile strength test on samples dried at 110° C, 4 weeks' rotting.

[1] C. P. Fiske, *Op. cit.*, p. 1722.

(6) Volume shrinkage on samples dried at 110° C, computing these data for comparison records.
(7) Water of plasticity.
(8) Water in terms of true clay volume.
(9) Shrinkage water in terms of true clay volume.
(10) Pore water in terms of true clay volume.
(11) Ratio of pore to shrinkage water.
(12) Apparent specific gravity.
(13) Porosity changes of clay briquettes, drawn at 850–1000° C and at each 50° C interval to 1450° C.
(14) Volume changes of clay briquettes drawn at above-mentioned temperatures.[1]

By following a complete set of tests, all the properties of a clay may be tabulated and preserved, and in a short time one will have certain comparable criteria established, whereby the suitability of a clay for retorts, glass pots or crucibles can be readily ascertained.

Preparation of the Batch.

In the preparation of the batch, the raw materials are ground, separately, in suitable machines, usually dry pans, to pass the required mesh screen. The size of opening for the clay should be 6-mesh and 8-mesh for the grog. After screening, the materials are elevated to storage until used. It is of extreme importance that the proper size of grain for the grog be selected. The grog forms the skeleton of the retort, and its function is to reduce the excessive shrinkage of the plastic bond clay and to regulate the evaporation of the mechanical water, thus helping to prevent the development of cracking during drying. The particles of plastic clays are supposed to be uniformly distributed around the grog grains, binding the whole into a compact mass which gives stability to the refractory retort. The grog also functions to absorb water in the pugging and to gradually return this moisture to the plastic clay during the drying, thereby making the rate of

[1] Procedures for these tests may be found in the Report of Committee on Standards, American Ceramic Society; also see Bleininger and Loomis, *Trans. Am. Ceram. Soc.*, **19**, 601–635 (1917).

evaporation as uniform as possible. As large-size grog particles as possible are to be desired for insuring the property of withstanding sudden changes of temperature to which the retorts are subjected during the transfer from the annealing furnaces to the smelting furnaces, and in the charging of the retorts with new loads. It has been found[1] that by using a judicious selection of grog sizes, and with the same bond clay, the tensile strength of a retort mixture could be increased 50.0 per cent and the porosity noticeably decreased, both being decided advantages. Fuller[2] advocates the use of parabolic formulas in which the diameters of the particles are the abscissa and the percentages are the ordinates. A factory combination of the various sizes of grog approximating Fuller's theoretical conditions consists of:

25 per cent through 10-mesh on 16-mesh,
25 per cent through 16-mesh on 30-mesh,
50 per cent through 30-mesh.

It has been the experience of many clay workers, when attempting to size their grog by mechanical operations, to be handicapped by the moisture variation affecting the operation of the screens. Provided a system of mechanical screening can be successfully developed, there is no doubt but that broader studies in the use of sized grog for all kinds of refractories to attain maximum strength and density will be forthcoming.

Proportion of Raw Materials.—In general, one can state that the clay content of a retort batch runs from 35 to 50 parts; usually 40 is the accepted figure, the remainder being grog. Sometimes the amount of clay is reduced by the addition of sand or coke, or both. The grog usually consists of all new calcined flint bodies or equal parts of new clay with old retorts or brick bats. The flint clay is rarely used raw.

The following commercial batches are presented for comparison:

[1] C. P. Fiske, *Op. cit.*, p. 1724.

[2] Concrete, Plain and Reinforced, Taylor and Thompson, chapter by W. B. Fuller.

	Per cent.	Per cent.	Per cent.
Plastic clay	46.1	40	36
Flint clay	47.8	50	54
Pulverized coke	6.0	10	10
	99.9	100	100

Methods of Mixing and Pugging.—The constituents of the batch, having been ground separately to the required size, are now mixed dry in the predetermined proportions, either by shoveling on a floor, or more commonly, by a mechanical weighing and mixing machine. From the machine the dry mix drops into the hopper of a horizontal pug mill, wherein the proper content of water necessary to develop the plasticity of the clay is added. This amount of water is usually from 8.0 to 10.0 per cent but may run as high as 20.0 per cent. As the clay issues from the mill in a semi-plastic state, it is cut off in large blocks weighing about 60 pounds each. These are loaded on to carts or wheelbarrows and taken to the rotting pits to age. The rotting pits are large brick chambers with no windows or other means of ventilation. Here the material is piled up and covered with damp burlap, and the room is closed off by sliding fire-doors for a period of 3–4 weeks. In this way the clay is allowed to develop its full plasticity. After the material has rotted or aged for the desired time interval, it is taken to the pug mill for another pugging.

It often happens that some of the pugs have been dried on the surface by air currents sifting through cracks in the walls of the damp room, and by re-mixing these pugs there will be obtained uniform working consistency throughout the whole mass of clay before it goes to the machine. During the process of this second pugging the mill operator adds a little additional water or dry retort batch, according to his sense of feeling whether the mix is too dry or too wet, before the pugs go to the presses. From the second pugging the clay batch passes into a vertical pug mill with a horizontal outlet. This machine forms the clay into ballots the correct size and shape to fit the presses. From here, the ballots are elevated to the press floor above by means of a ballot conveyor-elevator and are then ready to be made into retorts.

Before taking up the next step in the résumé, the writer would like to mention the increase and decrease in tensile strength gained by ageing some clays as reported by Fiske.[1] Data reported on this point show the following results for a St. Louis clay dried at 110° C:

24 hours' rotting, tensile strength	67.5 pounds per sq. in.
1 week's rotting, tensile strength	83.0 pounds per sq. in.
2 weeks' rotting, tensile strength	93.0 pounds per sq. in.
3 weeks' rotting, tensile strength	67.0 pounds per sq. in.
4 weeks' rotting, tensile strength	39.0 pounds per sq. in.

Evidently the development of the plasticity up to a certain point is beneficial, but it can be overdone. There is also some question in the writer's mind as to whether the same results would hold true on all refractory fire clays used in the refractory batches. A study of the effect of the temperature of the rotting chambers on the strength of the clays has been mentioned as worthy of investigation but no information along this line has been published to the writer's knowledge. The condition of too much ageing is seldom attained in retort potteries or glass-house refractories plants. Generally speaking, it is the other way. The clay mixes often used come directly from the pug mills with no ageing at all—a condition which one sometimes has to endure to keep the factory output up to the efficiency standard set by administration at the expense of the quality of the product.

The Manufacture of Retorts.

Forming the Retort.—The retorts are most commonly made by auger machines, but in this country some of the big plants[2] use hydraulic presses operating under a pressure of 1800 to 2000 pounds per square inch. The clay is quite stiff when introduced into the presses. This method gives a retort of superior quality: being much denser than the other type, it reduces the loss of zinc by absorption and filtration and increases the strength, rendering the product more able to resist the corrosion of the slags. These retorts are also thinner walled, thus providing a better rate of heat flow and the possibilities of longer service.

[1] C. P. Fiske, *Op. Cit.*, p. 1728.

[2] Mineral Point Zinc Company, Depue, Ill.

Press.—The hydraulic press used for forming the retort, according to Ingalls,[1] consists of a heavy steel cylinder, supported vertically by columns standing on a suitable pedestal. Operating inside of the cylinder there are two rams, one of them being annular and fitting to the main cylinder, the other being cylindrical, working inside of the annular ram. Pressure is exerted on both of the rams from below. The cylindrical ram being central to the machine, its own base serves as the piston. The annular ram is connected with a heavy yoke which is raised and lowered by two pistons working together, one on each side. The cylindrical ram passes through this yoke. Of the four columns which support the main cylinder, two diametrically opposite extend above the top of the latter; the other two extend only a little above the bottom, passing through lugs which are bored out for them. A heavy nut on the end of each column holds the cylinder down securely. The top of the cylinder is closed first by a heavy die block, the shape of the exterior of the retort, and above that a cover plate, both of which are hinged on one of the columns and can be swung horizontally aside by a lever; when in proper position they lock under the nut of the opposite column.

Operation of Press.—In making a retort, a ballot of clay is put into the cylinder from the top, both the die block and the cover plate being removed. The latter are then closed and locked, and both rams together are moved upward, pressing the clay solidly into the cylinder. The inner ram is then forced into the clay, while the annular ram, still under pressure, retreats as the clay is crowded backward around the inner ram. When the clay has been molded, the pressure is held for a minute or two, then it is released for an instant, and the cover plate is pushed aside, and the retort is thrust up and out through the die by the pressure raising the annular ram. The portion of the cylindrical ram which projects inside the die has the shape of the interior of the retort, and the bottom is formed between the ram and the cover plate.

One of these presses will turn out on piece work basis about 320 retorts in 8 hours when operated by a crew of four. A press

[1] "The Metallurgy of Zinc and Cadmium," **1906,** p. 239.

crew consists of four persons, *viz.*, the press operator, press helper, trimmer and trammer, of which two may be women, as is the case in the Mineral Point Zinc Company's plant at Depue, Ill. The press operator's duties are to fill the press with the ballots and to handle the pressure valves which control the press-plunger. After the vessel has been formed and pushed out, this workman cuts it free from the press with a piano-wire cutter.

The press helper swings on and off the die and cover plate and arranges the counter-weighted plate against which the retort is pushed out. As a part of his duty, he cleans out the die and greases it with press oil for the next retort.

As the retorts are cut off from the press, they are put in wood or light metal cases fitting over the lower half of the retort. It is the job of the trimmer to take one of these cases to the press as the finished retort is pushed out. The trimmer places the retort in the case and trims it to a length of 61.5 inches. The date is stamped on and the retort is now ready for the trammer who carts it to the dry room. The trimmer and trammer are oftentimes women.

Drying the Retort.—In a well-arranged retort pottery the dry rooms are on both sides of a long corridor. The rooms are accessible by car tracks running along the corridors in front of the dry room. To insure easy trucking away of the green and dried retorts, a turntable is placed in front of each room, thereby permitting a connection with a track extending the length of the room.

The drying rooms usually have a latticed floor, under which are located the heating coils to furnish the heat for drying the retorts. In the top of the room flues are located, to carry off the excess moisture from the green retorts and to insure circulation of air to expedite the drying. The rooms are closed off tight by sliding fire-doors when completely filled with retorts. The retorts are placed in the room by the trammer, starting at the back of the room first and setting the retorts on their butts close together. When the room is filled, it is closed off and allowed to dry from 50 days to 4 months, depending on operating conditions. The temperature maintained in the drying room is around

52–54° C. In the winter the first steam coil is put on for about a month, then the second one is turned on and left until the retorts are bone-dry. It has been found that the retorts nearest the walls dry more rapidly and show a strong tendency to crack and break. This is probably due to the strong air currents which develop along the walls of the room and to unequal drying conditions of the retorts; this same phenomena is noticed in glass-pot drying.

Storage.—When the retorts are dry they are loaded into trucks and carted to storage rooms, located usually in the furnace buildings quite near the annealing kiln. The retorts used for the next day are taken from the storage to the heating-up or annealing kilns to be burned and transferred hot to the zinc distillation furnaces. The great storage problem is due to the long time interval from the forming through to the drying period. This takes usually 120 days. Thus, in a plant operating 1500 distillation furnaces and having an average breakage of around 3.0 per cent, the total number of retorts required in storage stock all the time would be $1500 \times 0.03 \times 120$, or 5400 retorts. The problem of drying and storing is therefore no small item around the pottery.

Burning.—The burning of retorts is very similar to that of glass pots, except that the period of heat treatment is much shorter. The kilns are of both the up- and down-draft type; the better ones are very much like the modern pot arch and consequently require no detailed description. The number of retorts that must be burned daily depends upon the average breakage in the distillation furnace. This number can be estimated very closely from the records of past experience. The estimated number of retorts is transferred from storage to the kilns during the morning of the day before going into the distillation furnace. The kilns are fired up about noon, gradually at first, and, as the heat rises, the rate of firing is increased until by 5 or 6 A.M. the next day a temperature of 750–800° C has been attained. The work of filling and raking the distillation furnaces begins at 6 A.M. and continues up to 10 or 11 A.M.; during this time any defective retort is removed from the furnace and a new one from the annealing kiln is put in its place. The annealing kilns

are situated in several parts of the distillation furnace room, easily accessible to the furnaces, as the hot retorts are difficult to carry. The usual practice is to place one annealing kiln between two opposite distillation furnaces.

Condensers.—The condensers are not subjected to as severe heat treatment as the retort, they being located on the outside of the furnace proper. Hence, they do not require as good a grade of refractory clay and can be made faster and handled roughly. The condensers are formed by throwing a ball of condenser batch into a conical sheet-iron mold, a revolving water-lubricated pin boring out the clay forming the leg-shaped pipe of clay. After drying overnight, the condensers are crimped over a form, and this crimping causes a bellying out of the central part of the condenser. The crimped condensers are allowed to dry for 2 to 3 days, when they are burned to 900–1000° C. Some plants use as good a grade of clay for their condensers as for their retorts, but it has not been a general practice.

The life of a retort is generally from 30 to 40 days under severe treatment in the distillation furnace. Many break before this time, due to faulty structure and other causes. The number of retorts which have to be replaced daily runs about 3.0 per cent of the total number in the distillation furnaces. The loss of zinc through the walls of the retort by filtration and by absorption into the walls is quite a factor which the chief of spelter of the zinc plant would like to cut down. The machine-made retort and the glazed retort have in a measure overcome some of this loss. However, the absorption of zinc in the retort wall for the first seven days may run as high as 25.97 per cent the first day and as low as 1.7 per cent the seventh day, depending upon the density of the retort and the number of cracks developed therein. The amount of zinc left in old retorts may run from 6.0 to 10.0 per cent.

Mellon Institute of Industrial Research,
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A POSSIBLE CAUSE FOR THE DIELECTRIC FAILURE OF PORCELAINS WHICH ARE APPARENTLY FREE FROM MECHANICAL DEFECTS.

PRELIMINARY DISCUSSION.

By Chester Treischel.

From the first uses of porcelain as an insulation in high voltage transmission, difficulties have frequently resulted from the failure of insulators which visually appeared satisfactory, and electrical and ceramic engineers have endeavored to find the cause of these failures and an explanation of porcelain apparently losing its initial dielectric strength without apparent mechanical change.

One of the most widely accepted explanations of this breakdown phenomena is that analogy known as "hammering effect" of the current at high potentials. This analogy, however, becomes somewhat abstract when one asks for a definition of the term "hammering effect." It would seem then that a theory founded on the concrete instead of an analogy founded on the abstract would be a better explanation of electrical failure and would be of greater value to the scientist.

For a number of years it has been known that if crystals of certain substances, such as quartz, are subjected to a pressure on two diametrically opposite faces, these faces being parallel to the major axes, a measurable potential difference is set up, and that this potential difference varies directly as the pressure. This phenomena, known as the piezo-electric effect, was described by Lord Kelvin in two articles appearing in the *Philosophical Magazine*, 36 (1893).

The converse of this phenomena has been found to hold true, namely, that if a potential difference is set up on two opposite faces of a crystal, the sides being parallel to the major axis, the crystal is subjected to a squeezing action.

The application of such a phenomena in explaining the electrical failure of porcelain is at once apparent. Porcelain contains large

quantities of quartz crystals, this fact having been determined microscopically, the crystals being combined together by a glassy magma of fused feldspar. These crystals have no definite position with respect to the direction of their major axes.

Now then, what happens when we subject this porphyritic structure to the influence of alternating high potential? The theory of probabilities will uphold the statement that a number of these quartz crystals are arranged so that their major axes are at right angles to the direction of the flow of the electric current. The current itself is changing its polarity in accordance with the cyclic change, and during each cyclic change there are points of maximum and zero potential difference. If the current is of 60 cycles there will be 120 maximum potential differences and 120 zero potential differences per second. The effect on each individual crystal, which is arranged with its axis in the right direction, will be a vibratory movement parallel to the direction of the flow of the displacement current. This effect could cause breakdown of the dielectric in three different manners: first, a rupturing of the crystals, second, a rupturing of the glassy magma surrounding the crystals; and third, a leakage of current through the voids between the crystals and the magma when the crystal is at its greatest deformation and the potential difference is at its maximum.

RESEARCH LABORATORY,
GENERAL ELECTRIC COMPANY,
SCHENECTADY, NEW YORK.

DISCUSSIONS.

ALBERT PECK: While the writer is aware that in many minerals, possessing so-called polar axes, elastic deformation will set up a difference in potential at the opposite ends of the axes, and that conversely a difference in potential along a polar axis will elastically deform the crystal, he does not feel familiar enough with the phenomena to offer other than a few suggestions which might serve as starting points for further investigation.

In the first place, experiments of the type just mentioned are usually performed on perfectly developed crystals or those artificially produced by grinding. Quartz in porcelain is always present in irregular fragments and never in good crystals so that

if the above principle will not hold for properly oriented irregular fragments, the theory cannot hold. It would seem, however, that the outward form of the quartz is immaterial since we are concerned with directions only. While there undoubtedly would be a few fragments properly oriented with their major axes lying in a plane perpendicular to the direction of the flow of the current, there are also an almost infinite number of possibilities that the greater part are not oriented correctly. It, therefore, becomes a question of whether under the most favorable circumstances these properly oriented crystals would be present in large enough numbers to have sufficient effect to cause failure of the entire body. It is assumed that the author means that the vibratory effect gradually fatigues the fragments until complete rupture of the fragments results. If the fragments do shatter in this way, it seems possible that this could be detected by a microscopic examination of a thin section of porcelain which has failed—as we can similarly observe the shattering of quartz fragments in silica brick.

It is difficult to conceive of a decided change in specific gravity taking place in the quartz without inversion to some other form of silica. While contraction occurs along the axis in the direction of the pressure, there are also two other axes of like character and it seems probable that the contraction in the direction of one axis would be taken up by a corresponding expansion in the direction of the others and equally divided between them so that the volume would not change. Instead of conceiving of a greater density for the fragment, is it not easier to imagine that the fragment may contract in one direction and expand in others against the resistance of the feldspar bond, thus leaving space on one side where the current might leak through?

In passing, attention might be called to the effect of any heating which the porcelain may experience through induction or other electrical effects. Even slight increases in temperature may bring about electrolytic action through the feldspar matrix which rapidly lowers the dielectric strength and electrical resistance of the body.

Bureau of Standards,
Pittsburgh, Pa.

E. W. WASHBURN: Since the breakdown of the insulator occurs at comparatively moderate temperatures, it would seem to be improbable that any such explanation as that offered by the author is likely to correspond to the facts—owing to the extremely high viscosity and the corresponding difficulty of moving the crystals in the porcelain body.

If the explanation is correct, a microscopic comparison of the body before and after the breakdown ought to yield confirmatory evidence.

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URBANA, ILLINOIS.

W. R. GILDARD: In the burning of porcelain the flux attacks the quartz, taking a part of it into solution while the balance is more or less corroded, as may be seen by a microscopic examination in thin section. This solution is effected by what we may call "washing," firmly embedding the crystal in a matrix of varying silica content. As the crystal is being dissolved there is not the sharp line of demarcation as would be the case if the substance were insoluble (as an illustration, a piece of iron embedded in ice). It appears to the writer that the formation of a void of even microscopic proportions when the crystal is shortened is questionable, but rather that an additional stress is set up in tension.

The theory advanced appears to be an explanation of the "fatigue" and "hammering effects" of the electrical engineers.

According to the work of the Messrs. Curie and of Lord Kelvin, on this piezo-electrical property of quartz, the potential difference is sufficiently large to permit the truth of this proposed explanation being demonstrated in an experimental way.

RESEARCH LABORATORY,
GENERAL ELECTRIC COMPANY,
SCHENECTADY, NEW YORK.

SUBSEQUENT DISCUSSION.

CHESTER TREISCHEL: The uses of a theory, in the order of their importance, are:

1. It is useful in explaining facts as they are.

2. It is useful in forming a basis for the deduction of further facts.

3. It is useful in that it gives intellectual satisfaction.

Granting the above, what facts or phenomena will the theory of the vibration of the quartz crystals in porcelain explain? In what way might it form a basis for the deduction of further facts and for the carrying on of further experimental work?

To the writer it seems that this theory explains the following four fundamental facts:

1. That the substitution of certain substances such as talc, pyrophyllite, sillimanite, calcine, etc., for the quartz in an electrical porcelain, increases the dielectric strength of that porcelain.

2. That porcelains high in feldspar and low in quartz content are stronger dielectrically than those of low feldspar and high quartz content, the amount of clay used in either case being the same.

3. That porcelains tested with high frequency and with low frequency current at the same high potential, show that the disruptive effect of the high frequency current is much greater than that of the low frequency current.

4. That porcelains tested with direct or alternating currents at the same potentials show that the alternating current has a much greater disruptive effect than the direct current.

The replacement of the quartz in electrical porcelain with various substances has been attempted many times in an experimental way. In every case with which the writer is familiar, such a replacement has resulted in an increased dielectric strength. Even iron oxide in small quantities produced gratifying results. In this case, the amount of quartz present is materially reduced by the substitution of some substance for it. This substance being inert piezo-electrically, there of course is no crystal oscillation during the electrical test and the result is a porcelain of greater dielectric strength.

The same is true of the second case except that we do not have a complete substitution, but rather a partial replacement of quartz by feldspar. The piezo-electric effect, however, applies equally as well since there is a smaller number of quartz crystals present and consequently a smaller number of them properly oriented.

In explaining the greater disruptive effect of high frequency current over low frequency current we have in the case of the high frequency a greater number of pulsations per second than with the sixty cycle current. This no doubt fatigues the crystals more rapidly, causing a quicker rupture, just as in the case of the rapid and slow bending of a piece of steel we have the rapid bending causing the damage first.

With the use of direct current we have something of the same effect as that noted with the use of low frequency, the frequency here being zero. Of course, there is no vibration of the crystal in this case, merely a squeezing action—consequently a less violent effect upon the porcelain and noticeably greater dielectric strength than with the alternating current.

Now then, in what fields of further research will this theory lead us? It seems to the writer that there are two large and important ones, *viz.:*

1. The development of a substitute for flint which will not interfere with the proper working and burning of the porcelain batch. In other words, we want a material which has all of the good qualities of flint and enough inherent good qualities of its own to overshadow the bad qualities of flint.

2. The development of a feldspar substitute which will have all of the desirable fluxing and dielectric properties of feldspar but at the same time will be more elastic and less brittle.

THE TECHNIQUE OF OPTICAL GLASS MELTING.

By Clarence N. Fenner.

Without going into a full account of the circumstances which led to the coöperation of the Geophysical Laboratory with the manufacturers of optical glass of the country it will be desirable, as an introduction to this article, to summarize our activities in brief form.

Our coöperation began about the end of April, 1917, when certain members of the staff of the Laboratory were detailed to the plant of the Bausch & Lomb Optical Company at Rochester, New York. In December, 1917, the coöperation was extended to the Spencer Lens Company's plant at Hamburg, N. Y., and to the Pittsburgh Plate Glass Company's plant at Charleroi, Pa. At all these plants active coöperation continued for many months, and, with the last two, until the close of the war.

At the Bausch & Lomb plant the men from the Geophysical Laboratory had from the beginning the benefit of the many years' experience in glass-making of Mr. Victor Martin, chief glass-maker there, and the advantage of entering a works in which many of the difficulties attendant upon the first stages of starting up a new technical process had been surmounted. These facts were of the greatest assistance to us in becoming familiar with many details of practical glass-making, and further advances were thereby much facilitated. Although descriptions of ordinary glass-making operations were to be found in the literature, almost nothing had been published regarding the numerous important details in which the making of optical glass differs from that of other glasses.

At about the time that the work of the Laboratory was extended to the other plants, arrangements with the companies assumed a somewhat different form. At Rochester a more direct supervision of furnace operations was exercised; at Hamburg the Spencer Lens Company placed the entire operation and super-

intendence of its plant unreservedly in the hands of the Laboratory men; at Charleroi the supervision of the furnace-floor operations, in so far as the production and treatment of optical glass were concerned, was likewise placed in their charge.

The problems with which we had to deal were very different in many respects from problems of scientific research. Our task was essentially this: To produce as a matter of daily routine, glass in large quantities and free from serious defects; to seek constantly to eliminate defects and increase percentage of yield per pot; and to add to furnace capacity as rapidly as possible in order to meet the insistent demands for quantity production of good glass. The last requirement was always before us and practically everything else had to be subordinated to it. Problems of scientific importance, no matter how interesting, had to give way if they did not lead to the main object in view.

Because of the fact that the Laboratory men were at work at three different plants, among which conditions were, in some respects, quite dissimilar, experiences varied. By interchange of ideas this fact worked to the benefit of all. During the whole period under consideration the technique of the making of optical glass was in a state of change and development. New ideas which occurred to one or another were tested, and either rejected or, if found to be an improvement, were adopted as part of the routine of operation. From a consideration of this process of evolution it will not be a matter of surprise to anyone that the stage which has been reached at the present moment is not to be looked upon as the final stage of evolution, or that it is expected that no further progress will be made. It is fully realized that many problems of importance still remain for solution, but it is a source of great gratification to us that we have assisted in some measure in the production of thousands of pounds of optical glass of a quality equal to the demands imposed by military requirements, and much of it, as rigid tests show, not surpassed by the product of the more famous foreign works.

It would be beside the mark to describe the stages by which the present technique of optical glass melting has been evolved. It is rather the purpose to describe it at the stage which it has reached at present in its course toward more perfect development.

The description will be based not alone on individual experience but on the experience of all the men of the Geophysical Laboratory who took active part in this phase of the work. Those who were connected most directly with the operations which form the subject of this article were L. H. Adams, N. L. Bowen, C. N. Fenner, J. C. Hostetter, G. W. Morey, and F. E. Wright.

This article will deal with the melting process and closely allied operations. Particular attention will be given to those details of practice in which the melting of optical glass differs from that of other glasses, but in order to present matters in connected form it will be necessary to give an account of operations in the sequence in which they occur, even though this may involve some description of processes with which all glass-makers are familiar. It is not the purpose to emphasize any matters with whose development the members of the Geophysical Laboratory were especially concerned, but rather to present, for general information, a plain description of processes which have been found satisfactory in the making of optical glass. Other phases of the work connected with the preparation of optical glass will be treated by other writers in separate articles. Some of these have already appeared and others are being prepared at the present time.

Preliminary Operations.

An essential need in making good optical glass is pure raw materials. Especially important is freedom from those metallic oxides which impart color, such as iron, nickel, and manganese, and freedom from such impurities as sulphates and chlorides, which tend to produce milkiness or opalescence in glass. A full consideration of the requirements which the raw materials must satisfy will be given in detail in another article. It is sufficient at this point to say that the glass-maker must know beyond doubt what goes into his batch, and this can be done only by frequent chemical analyses.

It must be realized from the beginning and borne in mind constantly that, from the standpoint of the purposes for which optical glass is to be used, it has little in common with other kinds of glass. In order to serve its purpose an essential requirement is

that its optical constants should be definitely fixed and maintained. Other requirements of a similarly exacting nature must also be met, and it is possible to do this only by the most careful control throughout the process of manufacture. It would not be amiss to compare the precautions required in the making of a pot of glass to those which must be exercised in making an exact quantitative analysis.

The batch is mixed in a long, open box of such capacity that the whole may be shoveled from a pile at one end to a pile at the other in the process of mixing. It is best to use broad, flat wooden paddles or grain-scoops for this purpose, rather than iron shovels. The amount of iron which would be added to the batch by the wear of iron shovels is probably extremely small, but wooden paddles or scoops are as easy to use for the purpose of mixing, and it is better to avoid iron tools where possible if only to impress the workmen with a proper appreciation of the dangerous qualities of iron.

The batch must be so well mixed that any portion which is left over after filling a pot will not differ appreciably in composition from that which has been used. This will be secured by shoveling the material at least three times from one end of the box to the other with a turning and spreading motion. At some stage during this mixing, preferably after the first transfer from end to end, the whole should be passed through a sieve of four to six meshes per inch, which rests in a frame at one end of the batch-box.[1] This will remove nails, chips, and so forth, and will insure that no batch-material is left in a lumpy condition. If the scale of operations is large enough to warrant it, some form of mechanical mixer may be used. In any case care should be taken to see that the mixing of the batch is so thorough that any appreciable fraction does not differ in composition from the whole. After the batch has been mixed it may be left in the batch-box until it is used, if operations are conducted in such a manner that only a short time will elapse, or it may be stored in a bin. A discussion of questions of this kind, however, is rather apart from the purpose of this paper. They are matters of considerable

[1] It would really be better to put all materials through sieves before adding them to the batch, as well as to make a sieving at a later stage.

importance in works-management, but are not peculiar to optical glass-making.

Attention should be directed to one matter at this point—the care which should be exercised to keep different batches separate. Any confusion in this matter is likely to lead to a chain of serious consequences which will not be broken until all stock on hand of possibly contaminated batch has been thrown away, and all glass produced from it (which is not usable even as cullet) likewise disposed of.

In order to carry out completely the idea of avoiding contamination we should have at least three batch-boxes, one to be used for crown glasses, a second for flints, and a third for dense barium crowns. Moreover, it is well to sweep out the batch-box each time before use. It may seem somewhat trivial to emphasize apparently unimportant matters like these, but it is by paying attention to details of this kind and establishing a standard of careful procedure throughout all steps of manufacture that the qualities essential to optical glass can be maintained.

Considerable leeway is permitted to us in the matter of the form of combination in which a given ingredient of the glass is added to the batch, but in some cases compounds which, at first sight, might appear unobjectionable are found to be limited in usability by certain qualities which they possess. As a source of K_2O we have the carbonate, bicarbonate, and nitrate. The sulphate is out of the question because of its tendency to segregate as a layer of "salt water" on top of the melt and because it has the effect, even in small quantities, of inducing milkiness in some glasses.[1] KCl is objectionable for similar reasons, and also because of its volatility. Commonly, most of the K_2O is added in the form of carbonate, with a very appreciable percentage of nitrate (say 25 to 33 per cent of the alkalis in the latter form). The nitrate produces oxidizing conditions during the thermal dissociation and recombination of the batch ingredients and this is

[1] J. D. Cauwood and W. E. S. Turner, "The Influence of Small Quantities of Chlorides and Sulphate in Producing Opalescence in Glass," *Jour. Soc. Glass Tech.*, 1, 187 (1917). C. N. Fenner and J. B. Ferguson, "The Effect of Certain Impurities in Causing Milkiness in Optical Glass," *J. Am. Ceram. Soc.*, 1, 7, 468 (1918).

a highly desirable function. If it be used in large quantities, however, there is danger on this score: that at a temperature which is considerably below that at which there is active combination of the basic oxides with silica the nitrate will be decomposed into a highly caustic liquid which is likely to segregate from the rest of the batch and attack the pot. For this reason it should not be used ordinarily in quantities greater than those given. At Charleroi, however, the Pittsburgh Plate Glass Company use a pot of their own manufacture which seems especially resistant to this corrosive action. This, combined with the fact that large quantities of cullet are added to the batch, enables them to replace even the whole of the carbonate with nitrate. They are thus able to avoid the several somewhat objectionable features of the carbonate. The bicarbonate has been said by some to cause difficulty in fining, but we have used a considerable quantity of it in pots of medium flint and baryta flint without any observable deleterious effects.

In most glasses the replacement of K_2O by an equal weight of Na_2O has not a great effect on refractive index or dispersion. As the price of potash compounds is ordinarily greater than that of soda compounds and has recently been very much higher, the total substitution of soda for potash would offer decided advantages from that standpoint. Unfortunately it does not seem that this may be done without evil effects ensuing. The use of a certain amount of soda seems to be desirable, as it probably increases fluidity, but according to general opinion (for which there seems to be some basis) the use of large quantities tends to increase the color. The amount to which Na_2O may be used without injurious consequences varies with the composition of the glass. Frequently in the common glasses it may be used in amounts equal to those of K_2O, but if the glass has a tendency to show more depth of color than is desirable it is better to keep the Na_2O down. Such glasses include those high in lead or high in barium. In a baryta flint which was made regularly at Hamburg (a glass containing about 38 per cent PbO, 6.5 per cent BaO, and 3 per cent ZnO) the alkali used was wholly K_2O. When we tried a batch containing Na_2O to K_2O in the ratio 1 : 1.18 the color was distinctly increased. Other evidence of the color-

inducing effect of Na_2O has also been obtained. For this we have not been able to find a wholly satisfactory explanation. It does not seem likely that the soda itself gives color, but that the effect is secondary. Possibly a large quantity of soda causes greater corrosion of the pot and leads in that way to a larger amount of iron in the glass, or the same amount of iron may show up more in a soda glass, or the soda used may contain a small amount of sulphide or other reducing material which causes more of the iron to assume the ferrous form and therefore exert a more powerful coloring effect. It would be of considerable interest to settle this question, although the present commercial incentive arising from the abnormally high price of potash may partly disappear. A great deal of information on the relative quantities of K_2O and Na_2O used in the Schott works at Jena may be found in Zschimmer's tables.[1] However, a study of the syntheses appearing there gives very little insight as to the principles on which the relative amounts of K_2O and Na_2O used were based. They seem to be almost haphazard. A little later in this article batches for a number of typical glasses made in this country will be given, and these will show the amounts of Na_2O which we considered safe to use.

As a source of lead for flint batches either red lead (Pb_3O_4) or litharge (PbO) may be used. Red lead contains a little excess oxygen and might for that reason be considered preferable. Practically there seems to be no difference. After observing the results obtained from a great number of batches, some made with red lead and some with litharge, subjected to the same furnace conditions, we have not been able to see that there is any choice between them. It will simply be a question of purity of material and of price (calculated on the basis of lead content).

With most of the raw materials the size of grain has little effect on melting, and as long as the material is not lumpy the size of grain gives us little concern. With sand, however, the processes of combination and solution during melting are slow, and if unduly prolonged (as when the sand is coarse) the consequences are bad in several respects. The length of time which a run

[1] E. Zschimmer, in "Handbuch der Mineralchemie, herausgegeben von C. Doelter," article "Glas." Bd. I, p. 855.

takes is increased; there is more opportunity for liquation of the easily fusible materials, leaving the quartz floating on top; and slow melting is likely to mean poor fining. Therefore, quartz sand of coarse grain is undesirable. A very satisfactory sand which has been used largely at all three glass plants was of a size to pass through a 35-mesh Tyler standard screen.

The Use of Cullet.

In all optical glass making a large proportion of the glass in a pot is rejected because of defects of one kind or another. Thus a large quantity of glass which is not of optical quality but which represents considerable monetary value simply from the materials used in making it is likely to accumulate. The defects which have caused rejection are likely to be bubbles and striae. By putting this glass through the melting process a second time under proper conditions the percentage of saving in good glass from this cullet will probably be as high as in glass made from a new batch. We should, therefore, consider the manner in which the cullet may be used.

A first and very obvious requirement is that each type of cullet should be kept separate. Moreover, glass which is very stony or contains other defects which will probably not be removed in remelting should be rejected entirely. Milky or opalescent glass, however, which is otherwise suitable, may be used without fear. On the other hand, it must be remembered that certain properties of the glass will be changed each time it is remelted. It will take up more and more iron and other material from the pot, which will increase the color and change the index. Selective volatilization likewise will affect the index. These last-mentioned factors are likely to be the effective ones restricting the amount of cullet which it will be wise to add to a batch. It is quite feasible to make melts of cullet only, and indeed this has frequently been done, but there is likely to be some deterioration of the glass from standard quality under such conditions.

The changes consequent upon remelting will be different with the different types of glass so that no exact figures can be given as to percentages of cullet which we may add. Probably in heavy lead glasses the effects will be more marked than in other

common types. The standard of quality which is aimed at and the general care with which operations are conducted will likewise affect the question. If to a batch which is supposed to give a glass having $n_D = 1.605$ and $\nu = 37.7$ there is added 30 per cent of cullet which is supposed to be of the same type but which in reality has $n_D = 1.603$ and $\nu = 37.9$ we cannot expect to get exactly what we want. The essence of the matter is that there is no special trouble of operation arising from the use of almost any amount of cullet, but as for the amount that the manager will use, that will be decided by the way in which the properties of the cullet which he has on hand correspond to those of the glass he is trying to get.

For ease of handling the cullet should be broken up. This may be done simply with a hammer, but labor is saved and a better job is made by passing it through a jaw crusher. The jaws need not be set to crush very finely. We merely wish to get a shaly mass which can be shoveled without trouble. After this crushing the material should by all means be passed over a magnetic separator. A surprising amount of iron gets into the glass from the jaws of the crusher, but most of it is easily removed magnetically. In using the cullet it may either be mixed with the batch or filled in with it in alternating layers.

Setting the Pot and Filling in the Batch.

We will suppose that a pot of glass has just been removed from the furnace and we are about to start a new run. We will suppose also that the furnace we use is a reverberatory furnace operated on the regenerative principle. This is the type used by the Bausch & Lomb Optical Company and by the Spencer Lens Company. At the Charleroi plant of the Pittsburgh Plate Glass Company some of the optical glass is made in non-regenerative stackless furnaces of approximately cubical shape, heated by a blast, and some in large regenerative furnaces of twenty pots capacity each, such as are used for making plate glass. In all these types of furnaces the glass-making operations proper are essentially the same. The chief point of difference lies in the ability to control the conditions to which each pot is subjected, and in that respect the single-pot regenerative furnace seems to

offer considerable advantages. The description which follows will be based upon the operation of such a furnace. In many cases the portions of the description which are not applicable to the other types will be readily eliminated by the reader who is familiar with furnace operations, but where it seems desirable the description will be supplemented by an account of the modifications of procedure which are necessary in other furnaces. However, the endeavor will be to point out and emphasize the salient features in furnace conditions and operation which are desirable in making optical glass in distinction from other kinds of glass rather than to consider in detail the design and construction of furnaces which are necessary for the attainment of these results.

The first operation is to get the "siege" or floor of the furnace in good condition for the next run. This operation, however, is common to other kinds of glass-making and a description will be omitted. Likewise we shall pass over the transfer of the new pot from the arch in which it has been heating to the furnace, merely remarking that it is important to see that when the pot has been placed in the furnace it rests level on the siege. When these preparations have been finished the fire which, several hours before, had been shut off, is started again, and the furnace is brought up to melting temperature.

We will suppose that the previous pot was taken out of the furnace about the middle of the afternoon and that the operations referred to have been finished about four P.M. As will appear immediately, working conditions will often make it desirable to run on about this schedule, although this will not of necessity be the case. The furnace has become fairly cold, as no fire has been burning for several hours, and until the temperature is brought up again there is a hiatus in operations. Therefore the temperature is raised as rapidly as possible. Under the best conditions this will take considerable time, and if the gas pressure is not satisfactory seven or eight hours may be required to reach 1400° C. In some cases this may be more of a loss than is evident at first sight. Unless the plant be a very large one, employing great numbers of men at all hours of the day and night, the working arrangements will probably be such that pots will

be finished and taken out at about the same time each day. This will probably render it impracticable to base operations on anything but a twenty-four or a forty-eight hour schedule. Until recently forty-eight hours has been considered the normal length of time for completing a run. It was evident that the time could be shortened considerably, but unless it could be brought down to twenty-four hours there was not much incentive to curtail operations. Recent operations at Hamburg, however, have shown that in most cases it is perfectly feasible to work on a twenty-four hour schedule and produce just as good glass. To accomplish this it is essential that there should be no long delays anywhere. Therefore, the importance of having a good supply of gas and bringing the temperature up rapidly is evident. In doing this the beneficial effects of exposing the pot to a prolonged baking and sintering have to be sacrificed in some measure, but if by making this sacrifice the run is shortened to twenty-four hours the advantages are in favor of this procedure. At Charleroi the conditions are such that there has been no necessity of choosing between twenty-four and forty-eight hours. The optical glass plant is run in conjunction with an establishment having a very large output of plate-glass, and a large number of men are available on all shifts throughout the twenty-four hours. Other considerations exert their influence here, and the result is that the schedule aimed at has been twenty-seven to thirty hours in length. A description of the practical details of the twenty-four-hour schedule and the conditions under which it is applicable are given in the article by G. W. Morey in this number.

After the temperature has reached 1400° C, it is held there for about one hour in order to complete the baking of the pot and cause it to assume the dense condition which it must have to resist the corrosive action of the glass.[1]

A word may be said here about the means of determining temperatures in glass-furnaces. Proper temperature regulation is a matter of the very greatest importance in making optical glass, and an exact and reliable method of measurement is essen-

[1] Even higher temperatures (1430°–1440°) have been used with good results.

tial. The old method of regulating temperatures by the eye is hopeless. Undoubtedly some men who have had years of experience become remarkably proficient in this respect, but even the best have their off-days. Thermoelements are now much in use, but as shown in a previous article,[1] they are not very reliable for this work. An optical pyrometer meets the requirements much better, and is probably the most satisfactory instrument for glass-furnaces that we have. A detailed discussion of this matter and a description of the use of such instruments will appear later as one of the articles of the present series.

Although thermoelements are unreliable as standards, it is nevertheless very convenient to have each furnace equipped with one. Their changes of calibration are likely to be slow, and if the readings are controlled occasionally by an optical pyrometer, they may be used for ready reference.

The furnace having reached the condition described, the pot is glazed with cullet. The object is to have the pores filled with material of the same composition as the glass which will be made in the pot. If this be not done, but batch-mixture be the first material put in, the most fusible and at the same time most corrosive elements of the batch will enter the pores and possibly effect a disaggregation of the pot-material, which will subsequently lead to leakage or stones.

At least three or four scoopfuls of cullet are used, and it is best to pour it around the inside of the pot and allow it to run down to the bottom. The quantity should be sufficient to form a pool at the bottom, and if a large amount of cullet is on hand and is to be disposed of, it may be added in much larger amount than mentioned. The pot is then allowed to stand one hour longer. By holding the furnace for two hours at 1400° C before starting to fill in the batch proper we have made sure that all parts of the furnace and regenerative chambers are well heated and that the heat has penetrated the furnace lining to such a depth as to make available a certain store of heat for aiding in

[1] C. N. Fenner, "Methods of Temperature-Control in Glass-Melting Furnaces," *Phys. Rev.*, 2nd series, **11**, 2, 141. Abstract of a paper presented at the Rochester meeting of the American Physical Society, October, 1917.

a quick melting of the batch. The quicker the batch melts down the more satisfactory is the fining likely to be.

The normal quantity of batch to put in in the first fill is that which will about half fill the pot with unmelted batch. If the glass is one which is apt to give trouble in fining it is well to use a less quantity in order to give quicker melting, and fill in oftener.

When the first fill has melted down to a level surface, or in about three hours, a second fill of the same quantity is put in,

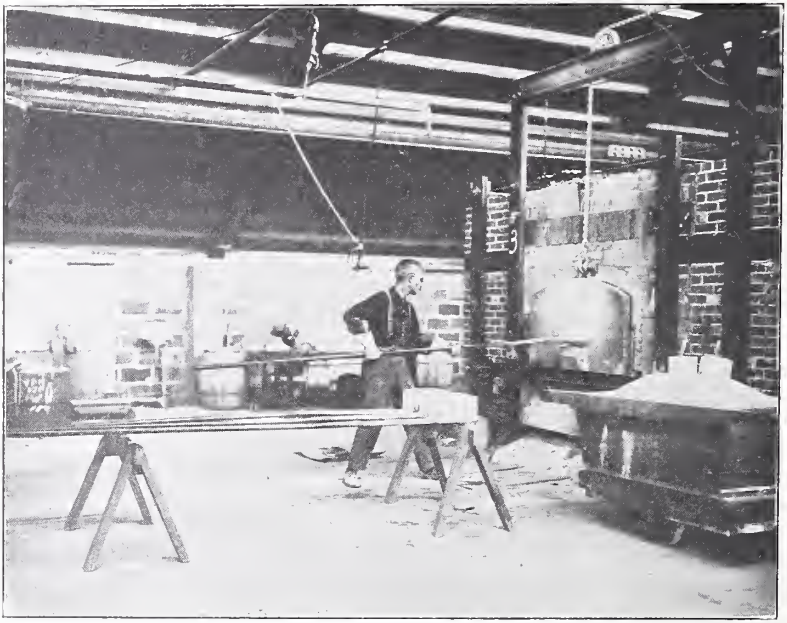


FIG. 1.—Filling in the batch.

and three hours later a third fill. Then one after another smaller fills are added, and finally the melt is topped off by a few scoopfuls of batch. The level at which filling ceases is that at which the melt is an inch or an inch and a half below the rim of the pot. The operation of filling is illustrated in Fig. 1.

Melting and Fining.

During melting such volatile substances as water, carbon dioxide, and oxides of nitrogen, likewise the excess oxygen in red lead, are driven off and the bases unite with silica (and with boric oxide, if that is present). A few moments after a fill is put in we may see little eruptions of gas, given off here and there from the batch, breaking through the liquefied surface. On the whole, however, the melting process is rather quiet. The presence of niter insures that the conditions shall be oxidizing, and gives the melt a fair start in the right direction in that respect. It is our business to see that the subsequent history does not involve reduction. That is a matter of keeping the furnace atmosphere in proper condition, and will be discussed later. Most of the volatile gases given off in melting escape entirely, but a relatively small amount is entrapped as bubbles, and another portion, there is reason to believe, is dissolved by the molten glass. The combination of the basic oxides with silica is likely to be slow, and as the silica is of relatively low specific gravity, this gives an opportunity for gravitative differentiation. In flint glasses especially a surface layer containing large quantities of grains of undissolved silica is likely to persist for a long time. In very dense flints a bottom layer of excessive lead content is also formed.[1] In one striking case which came under observation a pot of glass, which was supposed to have an index of 1.648, showed, because of improper stirring, the following phenomena: At the bottom a distinct layer, of about a foot in thickness, of glass of a pale greenish-yellow color, having an index of about 1.690; above this a mass of glass in which the index varied from 1.630 in the lower portions to 1.610 in the upper. During the melting of this glass samples from the top had shown quantities of silica grains embedded in a glass having an index of about 1.545. Probably in most cases a glass of this kind has undergone a similar segregation, but in this example the subsequent procedure was faulty and the segregation was not destroyed by stirring. Possibly, also, in this case the fills were not given time to melt down

[1] Liquation is naturally less pronounced when a large amount of cullet has been added before the batch is filled in.

sufficiently before others were added. In a paper by N. L. Bowen[1] phenomena of this nature are discussed in some detail.

It has been held by some who have made observations on gravity-separation effects in glasses that the evidence points to liquid immiscibility, but to anyone who has studied the phenomena of glass-making for a long period and who has been able to make observations at all stages of the process this explanation appears entirely unwarranted. Nothing seems more certain than that, if the mixing is sufficiently thorough, a homogeneous solution is formed, and that it is only by subsidiary processes, such as volatilization from the surface and solution of the pot walls, that there is a tendency for the glass to become inhomogeneous later. It is necessary, however, that such compounds as chlorides and sulphates be excepted, for the solubility of these in the melt is undoubtedly rather small. Instead of forming a homogeneous solution they rise to the top and form immiscible layers.[2]

The filling and melting will probably take eight to ten hours. Ordinarily this will be followed by a period of several hours during which the melt is allowed to stand and fine. However, according to the twenty-four-hour schedule it has been found possible in most cases to dispense altogether with fining as a separate process. The glass is given a hand-stirring before the melting is really complete. Then, a short time after the last fill, the machine-stirrer is set in operation and stirring continued until the pot is ready to come out.

During most of the fining period the glass is allowed to rest quietly. Filling has taken, we will say, from 10 P.M. to 8 A.M. Two or three hours later most of the surface of the glass should appear smooth and mirror-like, but if a proof or sample be taken the glass will be found to contain numerous large and small bubbles. Gradually the quantity will diminish. The large ones will disappear first. There is usually a period during which the evolution of large bubbles is quite active and this effect is termed the "boil." The fine bubbles ("seed") will continue rather longer

[1] N. L. Bowen, "The Significance of Glass-Making Processes to the Petrologist," *J. Wash. Acad. Sci.*, **8**, No. 4, p. 88 (Feb. 19, 1918).

[2] See F. Gelstharp, *Trans. Am. Ceram. Soc.*, **14**, 665 (1912).

than the larger ones but, if matters go properly, they should disappear almost entirely within a few hours. Occasionally a pot of glass will, for some reason, show a persistent layer of froth on top. One dislikes to see this, but nevertheless, such a pot will frequently come out all right in the end.

During fining the direction of flow of the air and burnt gases through the regenerative chambers should be reversed often enough so that one side does not become chilled and the other overheated. Reversal at intervals of twenty minutes answers very well for this.

For most of the common types of glasses a temperature of about 1400° C during melting and fining is required. If the batch materials contain sulphate or chloride in any but very small amounts and the glass is one which is likely to turn milky because of these impurities,[1] the danger may be avoided by keeping the temperature at 1420–1430° C during these operations.[2] This temperature is somewhat severe on the pot and the danger of the pot leaking or casting stones is increased, but pots are now manufactured in this country of such quality that, with proper care in previous treatment, the risk of disastrous consequences from running at this temperature is not so great that it need cause us to hesitate. It is essential, however, to see that, through carelessness, the temperature does not rise 15 or 20 degrees higher than the point established. Excessive temperatures are not only hard on the pot but increase the amount of selective volatilization from the surface and thus tend to alter the index of the glass.

Under ordinary circumstances (that is, when there is no danger of milkiness) a temperature of 1400° for a light flint or a boro-silicate crown answers very well. An ordinary light crown forms a more viscous melt and may be run at 1410°. Dense flints are very liquid, and need not be heated to more than 1370°.

Zschimmer[3] gives the following as the melting temperatures used at the Jena works:

[1] Light flints seem especially susceptible.

[2] C. N. Fenner and J. B. Ferguson, *Op. cit.*

[3] E. Zschimmer, *Op. cit.*, p. 865.

1000° C Phosphate glass ($3B_2O_3$, $70.5P_2O_5$, $12K_2O$, $4MgO$, $9Al_2O_3$, $1.5As_2O_3$, according to synthesis).

1100° Dense lead silicate glass ($20.8SiO_2$, $79PbO$, $0.2As_2O_3$—Syn.).

1130° Borate glass ($52.5B_2O_3$, $1SiO_2$, $1.5Na_2O$, $1.5K_2O$, $6ZnO$, $12BaO$, $16PbO$, $9Al_2O_3$, $0.5As_2O_3$—Syn.).

1320° Dense barium borosilicate glass ($14.5B_2O_3$, $39SiO_2$, $41BaO$, $5Al_2O_3$, $0.5As_2O_3$—Syn.).

1370° Ordinary flint glass ($45.7SiO_2$, $1.5Na_2O$, $7.1K_2O$, $45.4PbO$, $0.3As_2O_3$—Syn.).

1410° Ordinary crown glass ($2B_2O_3$, $69.2SiO_2$, $8Na_2O$, $11K_2O$, $4CaO$, $3.5ZnO$, $2PbO$, $0.3As_2O_3$—Syn.).

Where a covered pot is used, having only a comparatively small opening in front through which there is any possibility of furnace gases reaching the melt, the atmospheric conditions in the furnace around the pot are not of very great importance. Volatile materials, such as lead oxide, alkalis, and boric acid, are evolved and supply a protective covering within the hood of the pot, and through this protective atmosphere the furnace gases seldom penetrate. On the other hand, if open pots be used, it is important to keep reducing gases away from the glass. If the conditions within the furnace are kept anywhere near right the reducing action will never be sufficient to precipitate metallic lead, but at a considerably less intensity of reducing conditions the ferric oxide will be partly changed to ferrous oxide, and the same amount of iron in this lower state of oxidation has a much more intense coloring effect. In order to avoid this the gas should be burned with a rather short flame. A criterion which may be used is to see that the visible flames do not extend much beyond the top of the crown. Slowly moving flames, curling down over the pot, should be avoided if possible. In a multiple-pot furnace it may be difficult at times to avoid contact of the flames with the surface of the melt, but in single-pot furnaces, especially of the regenerative type, matters are under control to a greater degree, and here we may quite easily keep the temperature at the desired point and at the same time have the proper kind of flame by making correct adjustment between amount of gas, width of opening of air-inlet, and pull of stack, and a good furnaceman will soon learn to keep things very steady after having been shown what conditions are required. Some difficulty may be ex-

perienced at first in getting him to understand that economy in combustion may occasionally have to give way to other requirements.

Different types of glasses show considerable differences in the facility with which they free themselves of bubbles. For those glasses which give difficulty in this respect an old practice, which has been applied commonly not only to optical glasses but to other kinds, is "blocking." This consists in introducing some substances below the surface of the metal which will quickly evolve vapor. The large bubbles which are given off and rise through the metal tend to sweep out smaller bubbles with them. For this purpose various substances have been used—a block of wood, a potato, arsenic, etc. The Pittsburgh Plate Glass Company has developed the use of a substance which is especially applicable to optical glasses. A small amount of ammonium nitrate, sufficient to give rise to a number of large bubbles when it is inserted below the surface of the metal, is wrapped in paper so as to form a package. This is fastened to an iron rod which has been bent at almost a right angle, and plunged quickly into the metal and down to the bottom of the pot.[1] The ammonium nitrate is rapidly volatilized and the vapor rises in several great bubbles.[2] The blocking is begun when the metal has reached an open boil, at which time the operation is carried out three times in rapid succession, and fifteen minutes later three times more. This is continued until the metal is "plain" or free from bubbles. The fact that the vaporization of this substance is complete and without residue, and that the gas is oxidizing in character makes this material almost ideal for the purpose. Besides its effect upon enclosed bubbles it probably aids mixing somewhat by throwing up any heavy layer that has settled to the bottom.[3]

[1] We need have no hesitation in inserting iron tools temporarily in the melt if we see to it that their surfaces are kept free from scale.

[2] Instead of enclosing the ammonium nitrate in a paper wrapper it is sometimes melted over a low flame and cast in "pencils" in a mold. A section of one of these "pencils" is fitted into a cylindrical receptacle on the end of the rod and used as before. This method is rather neater and seems preferable, but it necessitates a little previous preparation.

[3] At Hamburg, in running on the twenty-four-hour schedule, it has been found effective in some cases, when the fine bubbles will not clean up, to stop stirring, turn off the gas, and drop the temperature from 1400 to 1250°, then turn on the gas, heat to 1400°, and resume operations.

The period of quiet during fining gives opportunity not only for the bubbles to rise but for pot-stones also to come to the surface. With a good pot the amount of stones cast should be small and often is negligible. If they appear they will be removed later by skimming.

Bubbles and Their Origin.

One of the chief purposes served in fining is the elimination of bubbles. It seems opportune, therefore, to discuss at this point the origin of bubbles, although such discussion will involve to some extent a reference to subsequent processes.

There are several obvious methods by which bubbles and seeds are formed, and others which are more obscure. Likewise the removal of some kinds of bubbles (or the bubbles in some glasses) gives no special difficulty, while in other cases the difficulties are very great. There are phases of this problem which have given us much concern and some questions regarding which we do not feel that we can give a definite and satisfactory answer. There are one or two types of glass which still give an occasional pot with many "seeds" (small bubbles) in spite of all precautions.

The great majority of the bubbles are doubtless entrapped mechanically, but others appear to be set free from solution in the glass. Of those entrapped mechanically many are to be referred to the first melting of the batch. With these the best possible opportunity for escape is offered during the period of fining, when the temperature is high and the glass is in its most liquid condition, and most of them should escape at this time, especially if assistance be given by blocking. We find, however, that often with the rather viscous melts, such as light crown, and less frequently with other melts, quantities of bubbles collect on the walls of the pot and adhere tenaciously. A proof taken after the first hand-stirring will often show many bubbles which have been set free by the agitation and have arisen to the surface, though prior to the stirring the glass may have seemed free of bubbles. These are probably to be attributed in part to gas which has been clinging to the walls and in part to gas which has been dissolved in the melt beyond the point of saturation and has

been set free by the mechanical agitation. In general, bubbles which have been entrapped during melting are not likely to be so troublesome as to cause us much anxiety. Certainly that is true with the more fluid glasses and probably with even the more viscous.

A second kind of mechanically entrapped bubbles are those which are stirred into the glass. During the last stages of stirring the viscosity of all glasses increases very greatly, and unless considerable care be used to adjust the speed of stirring to the viscosity the wave of glass which accompanies the stirring-tube is likely to fold over and entrap air. That this happens is not a matter of conjecture; such entrapped bubbles have been actually observed as they were swept downward into the melt. The remedy is to see that the stirring-tube does not travel so fast as to push a high ridge of glass in front of it. Further on in this article the stirring process will be described and data on the speed of movement for different types of glasses will be given. By using care bubbles of this kind may be avoided. Before the danger of entrapping air in this manner had been fully realized, a pot of borosilicate crown had, in one rather striking instance, been carried to a lower temperature (and greater viscosity) than usual. As a result the glass contained innumerable bubbles, whereas ordinarily this type of glass was almost perfectly free of them.

A third source of mechanical bubbles is a leaky stirring-rod. The stirring-rod is a water-cooled contrivance, at whose furnace end a reducing, right-angled elbow receives a hollow iron pin, which fits into the orifice left for the purpose in the upper end of the clay stirring-tubes (see Fig. 2). We have found that the joint made by screwing the pin into the elbow may appear water-tight when tested in the cold, but that during use in the furnace there may be a slow leak, probably due to unequal expansion. The evidence of this is twofold. First, we had the very direct evidence that when the pin was lifted out of the tube at the end of stirring a little dampness or even a drop or two of water has been observed on it. Second, while the stirring of the metal was being watched, a rather large bubble had been observed to rise to the surface of the melt occasionally, very close to the

stirring-tube. This had apparently been formed in the following manner: A drop of water had collected on the pin and had finally dropped down into the tube. There it had been vaporized so suddenly that some of the vapor had been forced out through the pores of the tube into the melt. If the melt were at a high temperature and of an intrinsically fluid nature the vapor rose to the surface, but if not it was distributed through the melt. An effective remedy for this is to set the pin in the elbow with a litharge-glycerine cement.[1]

In addition to the bubbles entrapped by the various methods which have been described it appears that there are bubbles of quite different origin. They seem to consist of gas which has been in solution in the melt at a high temperature and has been evolved on cooling. We have found that in some cases, after care had been taken to eliminate all the possibilities mentioned and the glass had become free from bubbles during fining, nevertheless, in the subsequent cooling during stirring, bubbles had reappeared and had persisted to the end. Such a phenomenon of greater solubility at higher temperatures is rather exceptional and is not, strictly speaking, one in which we have a simple solution of gas in a liquid. There is probably of necessity a chemical reaction between the gas and the constituents of the liquid. This reaction must be endothermic, for it is only under this condition that the solubility of a gas is increased on heating and decreased on cooling.

Our experience indicates that not all types of glass are affected by bubbles of this kind, and not any type under all conditions, but we are not able to state definitely what the conditions are which cause them to appear. We suspect rather strongly that those crown glasses which contain barium in small to moderate amounts are especially prone to dissolve gas and later evolve it as bubbles. Therefore, until more certain evidence is obtained,

[1] Made by adding litharge to glycerine and working it to a paste with a spatula. A very little water may be added to the glycerine. The time required for setting depends upon the fineness of grain of the litharge, amount of water, and proportion of litharge to glycerine. See a paper by H. E. Merwin in *Jour. Ind. Eng. Chem.*, **9**, 390 (1917).

we would look upon barium as a trouble-maker in such glasses and avoid its use where possible.

Still another class of bubbles, which may be termed vacuum bubbles, appear under certain exceptional circumstances. If a pot of glass be cooled quickly a rigid crust forms over the surface while the interior is still hot. Then when the interior cools and endeavors to contract it is held by the rigidity of the crust. If the glass contain bubbles of even the most minute dimensions (and probably they are always present), they act as points of weakness, and the glass contracts in every direction from them. Such a glass may, after cooling, appear full of large bubbles, especially in the last-cooled portions, but if it be carefully heated to a softening temperature the bubbles collapse until they are invisible or nearly so. If the proper procedure is followed, bubbles of this kind should never appear in our regular melts. They may be expected, however, in small experimental melts, where no special effort is made to have the glass cool properly.

The Function of Arsenic.

Closely associated with questions of the origin and elimination of bubbles is the matter of the use of arsenic in glass-making.

Arsenic in small quantities in the form of As_2O_3 is almost universally used in optical glass and very commonly used in other glasses. It is said to cause the melt to fine better and to make the glass more brilliant. The manner in which it accomplishes these results is rather uncertain. Allen and Zies,[1] in a recent article, have discussed the matter in the light of the latest information and have indicated the manner in which the oxides of arsenic may function in causing a boil and sweeping out the small bubbles, whose rate of ascent when unaided may be almost negligible.[2] We may amplify their idea slightly and add the suggestion that bubbles of arsenic vapors may not only gather up gases which have already been set free as bubbles, but that they may collect *potential* bubbles; that is, in the relation of these

[1] E. T. Allen and E. G. Zies, "The Condition of Arsenic in Glass and Its Rôle in Glass-making," *J. Am. Ceram. Soc.*, **1**, 787 (1918).

[2] Raymond M. Howe, "Principles Controlling the Formation and Removal of Bubbles in Molten Glass," *Trans. Am. Ceram. Soc.*, **19**, 201 (1917).

vapors to other substances which tend to be evolved slowly because of the hydrostatic pressure and surface tension[1] to which they are subjected, they may act as vacuum chambers into which these substances may evaporate at a much more rapid rate.[2] Although our knowledge of the manner in which arsenic acts upon the melt is rather deficient, the addition of arsenic is generally looked upon as being beneficial, and we may well continue its use. The amount added is always small. If an excess be used it has a tendency to produce a milky or opalescent glass. Zschimmer's tables show that at the Jena works the amount of As_2O_3 ordinarily put into the batch is about 0.3 per cent of the weight of the glass. In our own practice we generally used about the same amount or a little more.[3]

Leakage.—During the whole period of making a pot of glass a watch should be kept to see that the pot has not begun to leak. On account of the relatively corrosive properties of many optical glasses the danger is much greater with them than with plate glass, tableware, window glass, and batches of similar nature. The means which we found most effective in reducing leakage losses was to see that the temperature was kept uniform and not allowed to rise thirty or forty degrees beyond the established point as might happen through carelessness. The inauguration of a reliable system of temperature-control was attended by a remarkable difference in respect to losses of that kind. Inasmuch as the likelihood of leakage is greater with optical glasses,

[1] Raymond M. Howe, *Op. cit.* Howe shows that surface tension is probably a very effective force in restraining the growth of bubbles.

[2] According to the laws of gases a bubble filled with one gas represents, to all intents and purposes, a vacuum for a second gas. This is closely related to what is known as Dalton's law. See E. W. Washburn, "Principles of Physical Chemistry" (1915), p. 26, or W. Nernst, "Theoretical Chemistry," Fourth English Edition (1916), p. 39.

[3] The question of the use and function of arsenic requires further investigation. At Charleroi the preference was given to the use of considerably larger quantities of arsenic than 0.3 per cent, especially in the crown glasses. This practice was based on that of plate-glass making and the greater quantity was supposed to help fining. On the other hand, arsenic has been left out altogether in some instances at Hamburg, and the metal appeared to fine equally well.

precautions against it must be more strictly observed. In addition to keeping the temperature uniform other measures which should be carried out may be summarized as follows:

We must see that only best-quality pots are used; that they do not become damp or subjected to extremely cold temperatures while in storage; that they are handled gently while in the unburned condition; that the preliminary heating in the arch is done slowly and evenly and is carried to a bright cherry-red; that they are examined in the transfer from arch to furnace; that they are heated in the furnace to fining temperature or higher before filling is begun; that cullet is charged and allowed to soak into the pores before raw batch is added; and that for especially corrosive glasses the pot used be of a special clay mixture designed to withstand corrosion. With these precautions regularly enforced the number of pots that will fail will be extremely small.

Skimming and Stirring.—In running on a 48-hour schedule the melt or metal should be fine or plain by the evening of the day following that on which filling began. In fact, the fining may have reached a satisfactory stage before this, but unless we are endeavoring to hurry the process the melt may be allowed to stand quietly until about 7 P.M. Then, if it seems necessary, the surface should be skimmed. The indications are the presence of a scum of froth or stones on the surface. The stopper of the hole in the tuille is taken down and the skimmer inserted. This is a long-handled iron tool at whose further end a skimming blade is joined at right angles to the handle. The operation must be performed in such a fashion that the blade passes over the surface of the melt with a smooth, even stroke without bringing about such swirls or other disturbances as would cause surface material to be carried down into the melt, and at the end of the stroke the material must be gathered up on the tool and brought out. Some knack is required to perform these operations properly. If they are improperly performed surface material is forced down into the metal and does not soon rise again.

Care should be taken not to remove more glass than necessary. There has been gravitative segregation or liquation dur-

ing melting, and the composition of the surface layer does not represent the average composition of the melt. Therefore the greater the quantity of glass that is removed the more the average composition of the whole is changed. It would be better if we could avoid skimming altogether, but where it is necessary to skim, as is occasionally the case, we should see to it that an unnecessary quantity of glass is not removed. Some workmen will fall into this error through carelessness and some from excess of zeal.

After the metal has been skimmed it is allowed to rest for an hour and then the first hand-stirring is performed. Prior to this, however, certain preparations must have been made in the way of getting stirring-tubes ready. The stirring-tube is a thick-walled tube of clay whose length is about equal to the depth of the pot. For a pot 27 inches in depth we should have the tube about 26 inches long when dried and burned, keeping in mind that an appreciable shrinkage results from the latter operations. The outside diameter at top should be about 4 inches for a tube of this length, and should decrease to 3 or $3^1/_2$ inches at the lower end.[1] At the upper end there is a circular flange about an inch thick, which projects about an inch from the main body of the tube, and is used for holding the tube when the stirring-rod is withdrawn. After a tube has been fashioned it is set aside to dry for a week or two. Then while still soft (though firm) the central hole is modified in shape a little at the upper end. By means of a coarse file the circular section is changed to that of a square which is tangent to the circle, for a length of about four inches. The purpose is to have the shape of the hole conform to the shape of the iron holding-pin of the stirring-rod which enters it. This pin is square in section for about this distance at its upper part (where it joins the main rod) and is circular below this for a length of, say, three inches. The exact value of these dimensions is not important, except that the dimensions of the hole in the clay tube must conform to those of the pin. The purpose of the pin is to hold the tube in proper

[1] The thickness of tube used varies somewhat at different plants, and some latitude is allowable. The requirements are that the tube should be large enough to be an effective stirrer without being unwieldy.

position, and the square section of a portion of it prevents rotation during stirring.[1]

After the tube has been dried and the central hole shaped in the manner described, the tube must be burned. About an hour before one is to be used it is transferred from the salamander or from the arch in which it has been heating to the furnace and laid on the fire-wall to receive its final heating. A few minutes before stirring begins the tube is lifted off the fire-wall with an iron hook and gradually and carefully lowered into the melt and left floating there for a few minutes in an inclined position, with the flange at the upper end hooked over the lip of the pot. In a short time any bubbles that may have been given off will have risen to the surface. Then the water-cooled iron stirring-rod is introduced into the furnace, its pin is inserted into the hole in the tube, and stirring begins.

Just outside of the furnace the stirring-rod rests on a small grooved wheel[2] which turns on a horizontal axis primarily and whose shank is revolvable in a vertical socket secondarily. This permits the farther end of the rod to move continuously around a circle. Near the handles which the workman grasps the principal weight of the rod may conveniently be taken up by an iron hook attached to a cord, which passes around overhead pulleys to a counterweight. The hose connections are made near the handle and a constant stream of water is kept running through. As the water emerges from the outlet hose it should be appreciably warm but not hot.

The hand-stirring is carried on for 15 minutes at a time, at the rate of 24–28 revolutions per minute. The movement is a

[1] This description applies to the practice at two of the plants. At the third the construction of the stirring-rod is such that the pin which enters the tube is simply the terminal part of the main rod bent at a right angle and drawn down to a smaller diameter. Its cross-section is circular throughout. This construction is simpler, but unless very careful blacksmithing work is done the pin is likely not to fit closely in the tube and the tube may wabble during stirring.

[2] This wheel is supported on a length of channel-iron which passes horizontally across the front of the tuille and is, in turn, bolted rigidly (though temporarily) to the buck-staves of the furnace frame. The device is kept in position only during the period of hand-stirring.

combination of a circular and a vertical motion. At one portion of the circle the end of the tube is brought as near to the bottom of the pot as can conveniently be done without scraping, and at another portion it is lifted several inches. The chief purpose is to produce a thorough mixture of the metal; therefore in medium and heavy flints the stirring should be especially vigorous.

A proof taken after the stirring is over will often show that a quantity of bubbles have been detached from the pot-walls or set free from solution in the glass and brought to the surface. They should be of rather large size and should escape easily from the melt.

After the 15-minute stirring is finished the water-cooled stirring-rod is withdrawn from the furnace. The tube is left floating in the melt in the same position as when it was first introduced. Hand-stirring is repeated during the night at two-hour intervals. At about seven o'clock the next morning stirring by machine is begun. The stirring-machine (shown in Fig. 2) is an electric motor-driven device so constructed as to reproduce the circular and up-and-down motions described under hand-stirring, but to perform the operations in a smooth, even manner while rigidly following a prescribed course, and to continue it for hours. At one time or another several types of machines have been tried at the different plants, and from a comparison of their performances it becomes quite evident what requirements such a machine should possess.

The machine should be easily movable from place to place and should be capable of being quickly removed when stirring is over; therefore, it is provided with traveling wheels with offset sockets on the plan of the castors of a chair. When the machine is in position in front of the furnace the weight is taken off the wheels by elevating screw-posts. By means of these posts one may also adjust the height a trifle, but the principal adjustments for height should be elsewhere. It is essential to be able to change the speed of operation, and for this purpose we may either use a constant-speed motor in connection with step pulleys, or we may vary the speed of the motor by a rheostat and running-box. The latter arrangement is more expensive but considerably better. In any case we should be able to vary

the speed of stirring from about 28 to 4 or 5 revolutions per minute, the variation being either continuous or in not less than four steps. The horizontal rotating arm to which the outer end of the stirring-rod is attached and by which a circular motion is given to both the outer and the inner (or furnace) ends of the rod, is mounted on a vertical axle driven by bevel-gears from the pulley-shaft. In order to attach the stirring-rod to the arm the

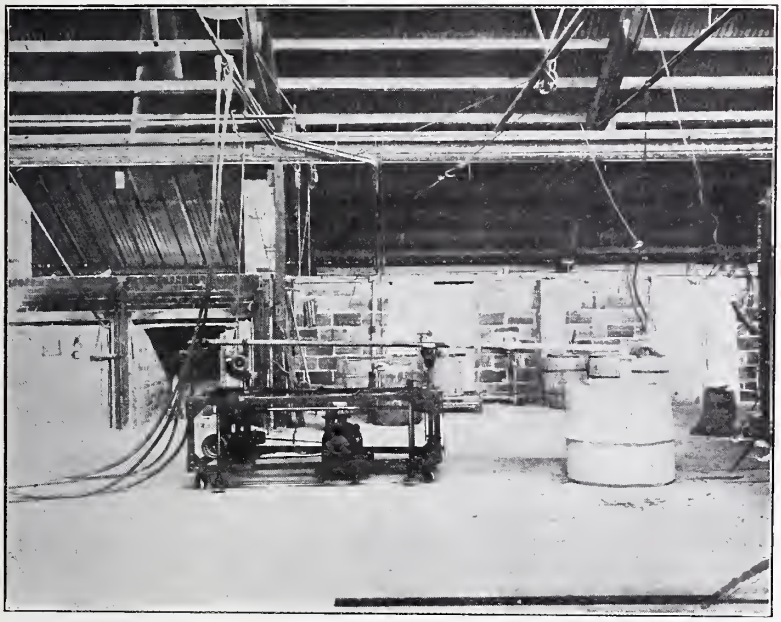


FIG. 2.—Stirring machine set up to show its position with relation to the pot.

latter is provided with a slot which receives a corresponding lug on the stirring-rod. A stout pin passes through the two and is held by a split cotter. This arrangement permits the rod to be mounted or dismounted in a moment. The height of the arm should be adjustable, and to effect this its attachment to the vertical axle which turns it is by means of a movable collar which may be slipped along the axle and held at the desired height by a set-screw.

The stirring-rod is supported at the middle on a grooved wheel similar to that used for the same purpose in hand-stirring, but here the shank (pedestal) of the wheel is extended to form a long rod or post, which passes through bearing-boxes on the framework of the stirring-machine and is attached to the mechanism in such a manner that an up-and-down motion may be imparted to the post and wheel by a crank-drive. The result of these various mechanisms is that the outer end of the stirring-rod is carried around a horizontal circle, the median portion is given a vertical motion, and the inner end combines the two. Strictly speaking, the horizontal component of the motion of the inner end is not circular. The curve described is an oval, which, however, does not differ greatly from a circle. The height of the wheel-post may be varied in a fashion similar to that described for the rotating arm. Likewise the length of stroke of the post may be varied, although this is hardly necessary. Some machines which we used were provided with means for varying the vertical stroke, but in practice they were left set at a stroke of about $2^1/_4$ inches. By all means, however, it should be possible to take off the vertical motion altogether with little trouble or delay. It is essential also that the effective radius of sweep of the horizontally rotating arm may be changed easily by a screw-feed. During most of the period of stirring the radius of the stirring-circle is left without change, but as the glass cools and stiffens in the final stages of stirring the radius of the stirring circle is cut down. The vertical motion of the supporting post should be related to the horizontal motion of the rotating arm in such a way that they do not synchronize but that the uppermost (or lowermost) part of the stroke advances a certain amount each time on the horizontal circle. A satisfactory arrangement is to have one complete stroke (from upper limit to lower limit and back) in about $1^1/_4$ revolutions. The whole machine should be rigid in construction and not likely to get out of order, and the settings and changes required for proper stirring should be easily effected.

When the machine is first set in motion the radius of the stirring-circle is cut down to the lowest limit so that the clay tube makes only a very small circle around the center of the pot. Then the circle is increased in size until it approaches the outer cir-

cumference. Care should be taken, however, to see that the clay tube does not approach the pot-walls so closely that it touches at any point or does not pull off the skin of glass adhering to the walls. For a pot whose diameter at bottom equals 25 inches we have found that a stirring-circle of 7 inches radius is a quite satisfactory maximum. As the stirring-tube has a radius of about two inches the circle passed through by the outer portions of the tube has a diameter of 18 inches under such conditions. At the speed with which the machine is driven all the glass in the pot is effectively agitated and mixed when the tube describes such a circle.

In setting the tube for height the endeavor is made to have the bottom of the tube come near enough to the bottom of the pot so that no layer of glass is left unstirred, but to avoid the danger of letting the tube touch the bottom. To accomplish this a clearance of about two inches between the end of the tube and the bottom of the pot should be allowed.

It is only with glasses which have a strong tendency to form a heavy bottom layer that a vertical motion is given to the tube in machine-stirring. Therefore, in general, the vertical motion is used only with medium and heavy flints. For these the vertical stroke of the supporting post may be made $2–2^1/_4$ inches. As this motion is imparted to the stirring-rod at midway of its length, the far end of the rod with its attached tube is given a vertical motion of $4–4^1/_2$ inches. It has been found in some cases that even with dense flints ($n_D = 1.650$) the vertical motion may be dispensed with. In these cases large quantities of cullet (40 to 50 per cent) were used, and under such circumstances there is much less likelihood of the settling of a heavy layer to the bottom.

The viscosity of different glasses varies considerably at the stirring temperature, and the speed of stirring likewise varies to some degree. Of the common glasses dense flint stands at one extreme and light crown at the other. A speed which has been found to work well with flints is 18 revolutions per minute with a stirring circle of 7 inches radius. This gives a speed of 792 inches or 66 feet per minute as the rate of horizontal travel of the stirring-tube through the glass. In addition there is, with

flints, a vertical motion imparted to the tube, as already stated. In the case of light crown and similar glasses the danger of stirring bubbles into the melt makes it desirable to run more slowly Therefore a speed of about 45 feet per minute has been used. For borosilicate crown the speed has been about the same as for flints.

Up to the time that stirring by machine has been begun the temperature has been kept up to 1400° C or thereabouts (varying somewhat with the type of glass). During the progress of machine-stirring the stopper is out continuously and the furnace naturally cools somewhat. This is quite allowable at this stage, but the drop should not exceed 75–80° C.

Machine-stirring is continued without change for four or five hours. Then the gas is shut off, the air-inlet to the regenerative chambers is closed, the stack-damper lowered, and the butterfly-valve swung to the intermediate position. The metal has been thoroughly mixed and now it is allowed to cool and stiffen. Stirring must be continued, however, for the sources of inhomogeneity are still active. The principal source is doubtless the bottom and walls of the pot, by whose solution foreign material is constantly supplied; but another source is the upper surface of the metal, from which selective volatilization has been going on. The actual change in mass composition due to these gains and losses is probably so small that when mixture is complete and uniform the optical properties are not greatly affected thereby. It is not, indeed, the change in total composition which gives us concern, but the likelihood that material of different composition will be only imperfectly distributed through the mass. It is astonishing to find what slight differences in refractive index are exhibited by cords or striae which are so pronounced as to stand out in plain relief. When the difference is only in the third place of decimals a cord is quite perceptible. This means that the difference in composition is so small that diffusion is not a very effective factor and must be mechanically aided. The principle on which stirring effects its results is that these heterogeneous portions of the melt are mechanically spread out to such a degree that they present great surfaces of contact per unit of volume and only a short time is required for diffusion to overcome all

differences that exist. From the time that the gas is turned off the procedure followed is directed chiefly toward eliminating the conditions which permit striae to form. To effect this the heterogeneous material must be thoroughly distributed through the mass almost at the source from which it proceeds; at the same time conditions must be so changed that the tendency toward solution of the pot and volatilization from the surface is decreased, while the mechanical obstacles presented by viscosity to the movement of convection-currents are gradually becoming greater. These objects are attained more or less perfectly by continuing stirring as vigorously as may be without running the risk of causing air to be entrapped, while cooling the melt to a temperature which has been found by experience to be suitable. This temperature varies widely with different glasses, and the time required for cooling varies accordingly.

If one watches the stirring while the temperature is dropping one sees that the ridge of glass pushed ahead of the stirring-tube gradually becomes more prominent and if no change in the speed of stirring were made the ridge would, before long, reach a height of several inches. Obviously it is necessary to decrease the speed as the glass thickens. The vertical motion of the tube is unnecessary after the fire has been turned off and may be dispensed with at that time, and soon it is advisable to lessen the speed of angular rotation and to diminish the size of the stirring-circle. The proper amount of diminution is judged by the appearance that the passage of the tube through the glass presents, and is a matter that can hardly be described but must be learned by experience. However, temperature charts and records of operations during some typical runs are here presented, which may serve as a basis of comparison. The information is taken without modification from records made during actual operations. Where it is believed that the procedure might have been improved at some point a memorandum is introduced. The radii of stirring circles given are those measured at the outer end of the stirring-rod, but as the point of support or pivot of angular movement of the rod is about midway of its length the same figures apply without much modification to the inner end.

EXAMPLES OF PROCEDURE IN STIRRING.

(See also Charts 1–4.)

No. 1. Medium Flint. (n_D = 1.605, ν = 37.6.)

Pot 25 inches in inside diameter at bottom, 27 inches deep.

8 : 03 A.M. Started stirring machine at 13 rev. per min. Radius of stirring circle = $6^1/_8$ inches. Vertical motion = $4^1/_4$ inches. (Mem.: A stirring circle of greater radius would have been preferable.)

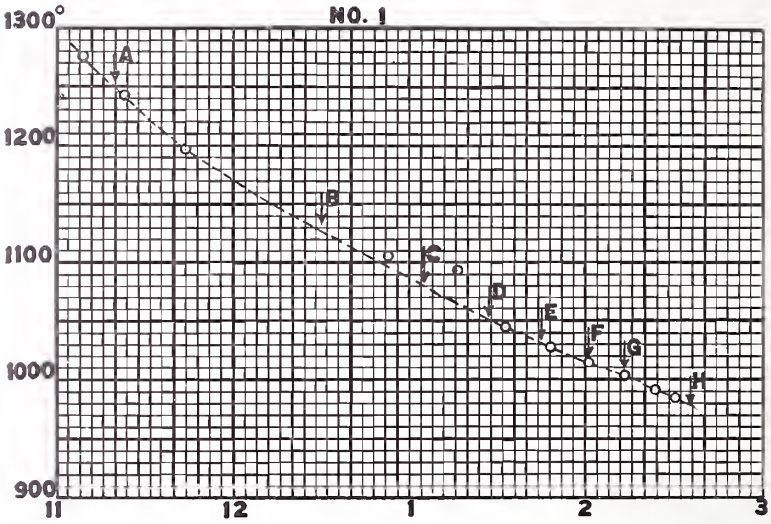
8 : 07 A.M. Changed speed to 18 rev. per min.

11 : 00 A.M. Shut off gas, air, etc.

11 : 09 A.M. Temp. (sighting on surface of glass) = 1276° C.

11 : 20 A.M. (A) Took off vertical motion.

11 : 23 A.M. Temp. = 1241° C.



11 : 44 A.M. Temp. = 1197° C.

12 : 30 P.M. (B) Radius of stirring circle reduced to $5^1/_8$ inches. (Mem.: It might well have been kept a little larger.)

12 : 53 P.M. Temp. = 1104° C.

1 : 05 P.M. (C) Changed speed from 18 to 13 rev. per min.

1 : 17 P.M. Temp. = 1092° C.

1 : 27 P.M. (D) Radius of stirring circle reduced to 4 inches.

1 : 33 P.M. Temp. = 1044° C.

1 : 45 P.M. (E) Reduced radius of stirring circle to $3^1/_4$ inches.

1 : 48 P.M. Temp. = 1028° C.

2 : 01 P.M. (F) Temp. = 1013° C. Reduced radius of stirring circle lightly.

2 : 13 P.M. (G) Temp. = 1003° C. Radius of stirring circle now about $1^1/_2$ inches.

2 : 23 P.M. Temp. = 990° C.

2 : 30 P.M. Temp. = 984° C.

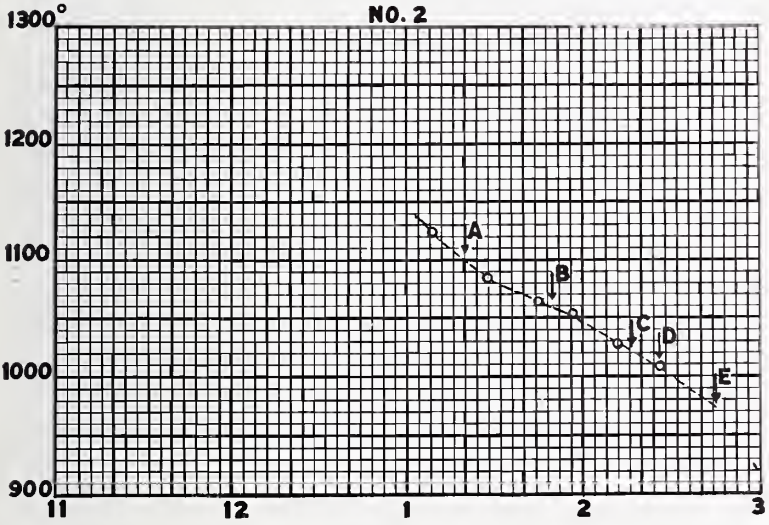
2 : 35 P.M. (H) Stirring stopped. Extrapolation gives temperature of 975° C.

2 : 40 P.M. Pot out.

(Mem.: A temperature of 950° C at time of stoppage of stirring would have been preferable for this type of glass.)

No. 2. Baryta Flint. (n_D = 1.619, ν = 37.6.)

Pot 20 inches in inside diameter at bottom, 25 inches deep.



8 : 15 A.M. Started stirring machine at 13 rev. per min.; soon afterward changed to 26 rev. per min. Vertical motion $4^1/_2$ inches. Radius of stirring circle = 5 inches.

11 : 50 A.M. Shut off gas, air, etc. Changed speed to 18 rev. per min. Took off vertical motion.

1 : 09 P.M. Temp. = 1122° C.

1 : 20 P.M. (A) Changed speed to 13 rev. per min.

1 : 28 P.M. Temp. = 1084° C.

1 : 45 P.M. Temp. = 1061° C.

1 : 50 P.M. (B) Reduced radius of stirring circle to 4 inches.

1 : 57 P.M. Temp. = 1053° C.

2 : 12 P.M. Temp. = 1028° C.

2 : 17 P.M. (C) Radius of stirring circle reduced to 3 inches.

2 : 26 P.M. (D) Temp. = 1007° C. Radius of stirring circle reduced again.

2 : 45 P.M. (E) Stirring stopped. Extrapolated temperature = 975° C.

2 : 50 P.M. Pot out.

No. 3. Light Crown. (n_D = 1.516, ν = 60.0.)

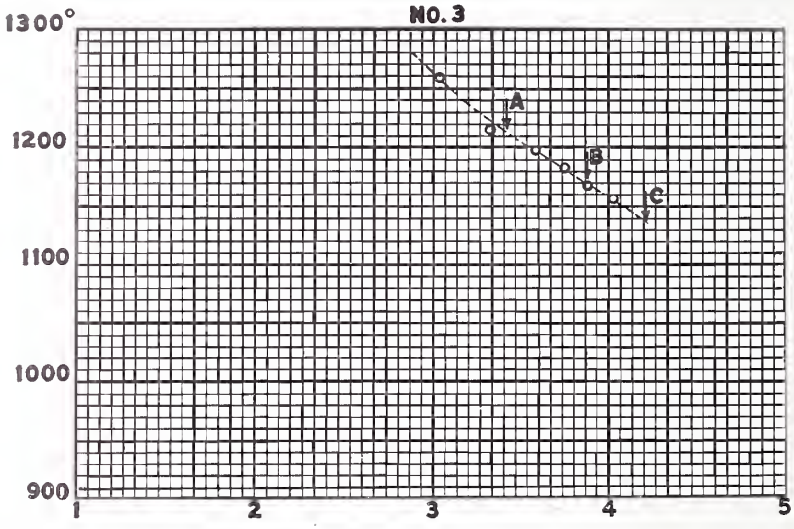
Pot 25 inches in inside diameter at bottom, 27 inches deep.

8 : 03 A.M. Started stirring machine at 13 rev. per min. No vertical motion. Radius of stirring circle = $6\frac{1}{2}$ inches.

8 : 25 A.M. Changed speed from 13 to 18 rev. per min.

8 : 55 A.M. Changed speed from 18 to 13 rev. per min.

9 : 50 A.M. Reduced radius of stirring circle to about 5 inches. (Mem.: A proof taken just before this had shown quite numerous bubbles; therefore,



the speed of travel was reduced to avoid the danger of stirring air into the metal.)

2 : 35 P.M. Shut off gas, air, etc.

3 : 02 P.M. Temp. = 1259° C.

3 : 19 P.M. Temp. = 1214° C.

3 : 25 P.M. (A) Radius of stirring-circle reduced to 4 inches.

3 : 35 P.M. Temp. = 1197° C.

3 : 45 P.M. Temp. = 1182° C.

3 : 53 P.M. (B) Temp. = 1168° C. Radius of stirring-circle = $2\frac{1}{2}$ inches.

4 : 02 P.M. Temp. = 1156° C.

4 : 13 P.M. (C) Stirring stopped. Extrapolated temp. = 1138° C.

4 : 18 P.M. Pot out.

No. 4. Borosilicate Crown. (n_D = 1.511, ν = 64.1.)

Pot 25 inches in inside diameter at bottom, 27 inches deep.

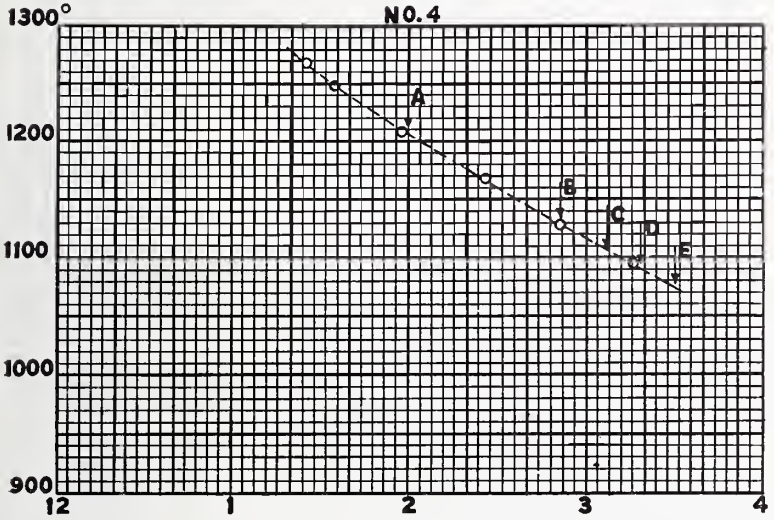
8 : 10 A.M. Started stirring-machine at 13 rev. per min. No vertical motion. Radius of stirring circle = $6\frac{1}{2}$ inches. Changed in a few minutes to 18 rev. per min.

1 : 15 P.M. Shut off gas, air, etc.

1 : 25 P.M. Temp. = 1266° C.

1 : 35 P.M. Temp. = 1247° C.

1 : 58 P.M. Temp. = 1208° C.



2 : 00 P.M. (A) Changed speed of stirring to 13 rev. per min.

2 : 26 P.M. Temp. = 1166° C.

2 : 51 P.M. (B) Temp. = 1127° C. Reduced radius of stirring-circle to 5 inches.

3 : 07 P.M. (C) Reduced radius of stirring-circle to 4 inches.

3 : 16 P.M. Temp. = 1095° C.

3 : 18 P.M. (D) Reduced radius of stirring-circle.

3 : 30 P.M. (E) Stirring stopped. Extrapolated temperature = 1075° C. (Mem.: This is rather high. 1050° C would have been preferable.)

3 : 35 P.M. Pot out.

Probably the examples given may be taken as fairly representative of the procedure to be followed during cooling and stiffening, but in making application to other cases caution must be used because of the fact that a type of glass having certain optical

properties may vary in chemical composition, and the treatment necessary will be closely related to the latter. For instance, it was found at Hamburg that the light flint made there should be cooled to 925° C before stirring was stopped. A glass of very similar optical properties but of somewhat different chemical composition (lower in alkalis), made at Charleroi, was removed from the furnace at a temperature higher by 100°.

There is much room for the personal equation to enter as an important factor in making measurements with the optical pyrometer on a pot of cooling glass. Different observers differ considerably in their ability to match the luminosity of the filament with that of the object sighted upon. Moreover, the angle at which the pyrometer is directed upon the surface of the glass is probably of some importance (because of surface reflections); and also, in the later stages of cooling, there actually are marked differences of temperature at different parts of the surface. At high temperatures the conditions of temperature-measurement are probably almost ideal for the employment of an optical pyrometer, as the light emanates from a self-luminous, transparent source within the mass, and the agreement with black-body conditions should be practically perfect[1]. Moreover, at this stage the temperature-differences in different regions are small. At a later stage, however, it is easy to perceive that the swirls of liquid brought to the surface by the passage of the stirring-tube are hotter than the surrounding material. Then the measurement of temperature becomes, to a certain degree, an empirical opera-

[1] This is true unless glass at a high temperature possesses the property of luminescence; that is, that it emits light whose intensity does not correspond to that which a black body would emit at the same temperature. This is a possibility which must be taken into account until our data on the matter are more complete. At present the probabilities seem to be against it.

If one tries to look into a pot of glass which has just been removed from the furnace and has a temperature somewhere between 950° and 1100°, the impression produced is not that of looking deeply into a transparent mass but of looking into something which has the appearance of a milky, luminous haze. The explanation is probably this: that a body whose luminosity is due to its temperature tends to cut off light of any wave-length which it itself is sending out. Therefore, in such a pot of glass, the cool crust is transparent to the light proceeding from the glowing mass below, but the latter is (within a certain distance) opaque to its own radiations.

tion, and one must decide where he shall sight the pyrometer. The temperatures which have been given and others which will appear in a similar connection later refer to the hottest parts of the melt, close to the stirring-tube.

The temperature at which stirring is stopped and the pot set out is a matter of the greatest importance. Upon this principally will depend whether the glass is badly affected by striae or not. There are probably other factors to which, at times, striae are due. One of these is failure to break up and diffuse by vigorous stirring the layer of heavy glass which has segregated and sunk to the bottom of the pot, especially in dense flints. The formation of this layer has been referred to a number of times, and attention has been called to the procedure to be followed to eliminate it. Striae may be caused also by setting the pot, after removal from the furnace, into an arch that is too hot, thus causing convection-currents to be set in motion. This also may be quite easily avoided by proper attention. It is a somewhat more delicate and difficult matter to have stirring stopped at just the right moment and have the pot brought out promptly.

On a previous page the principal source of striae has been traced to dissolved pot material. If stirring be stopped at too high a temperature, this solvent action is still going on quite vigorously, and the viscosity of the metal has not yet become sufficient to oppose much resistance to the rise of convection-currents. The hottest part of the pot is the bottom, for during cooling the siege has been protected by the pot itself. Therefore, during the few minutes which elapse after stirring has stopped and before the pot is lifted from the siege convection-currents tend to be set in motion from this source. For this reason the metal must be cooled to a point where this tendency is small. Moreover, no unnecessary delay should be allowed to occur between the stoppage of stirring and the removal of the pot. It might seem that cooling and stirring should continue to a point where the danger arising from convection-currents is obviously past, but this may not be done. There would be a probability of stirring air into the glass under such a procedure, and also a probability of dragging off into the melt the skin of glass lying next to the sides and bottom of the pot. This is a thin layer whose composition

is affected by solution of pot-material, and which, under ordinary circumstances, adheres closely to the pot, but may be pulled off if the stirring be continued until the glass is very stiff, and then gives rise to heavy cords. For these reasons the optimum temperatures for the cessation of stirring must be determined within rather narrow limits. Especially is this true with medium and heavy flints, whose compositions and optical constants differ greatly from those of the materials arising from pot-solution. With ordinary crown and borosilicate crown there is much less difference in this respect and more leeway is permissible.

In order to insure that the pot is set out at the proper temperature the fall of temperature is followed with the optical pyrometer during the later stages of stirring, and a few minutes before the end an extrapolation of the course of the temperature-time curve is made to the desired point. Following is given a list of temperatures for the cessation of stirring which have been found applicable to a number of common types of glass. In making use of this table the caution must be kept in mind that each figure applies to a glass of a certain chemical composition and not to a glass of a certain optical type. Moreover, it is not unlikely that furnace construction affects the rate of cooling, and perhaps other factors of an unknown nature may change the figures slightly. They may be taken at least, as fairly definite starting-points for experiment, and it is believed that, on the whole, not much change will be found necessary.

TABLE 1.

TEMPERATURES AT WHICH TO CEASE STIRRING.

(The compositions of the glasses to which these figures apply are given in Table 2.)

Borosilicate crown	1050° C
Light crown	1150° C
Baryta flint	980° C
Medium flint	950° C
Light flint	925° C
Barium crown	1025° C

During the cooling and stiffening of the glass the speed of stirring has been reduced and the size of the stirring-circle has been diminished, until, in the last stages, the stirring-tube re-

volves slowly around a small circle in the center. Then, at a moment which has been determined some time before, the electric current is shut off from the stirring-machine and preparations are made to take the pot out. By all means this should be done quickly, in order to prevent the rise of convection-currents from the bottom, and each workman who is to take part should know what his duties are. The first operation is to move the stirring-tube from the center over to the left side of the pot. This is done, before the stirring-tube is disengaged from the rod, by swinging the rod outward from the center by means of the screw-feed. This imparts a smooth, even motion to the tube and obviates the likelihood of its digging into or otherwise disturbing the layer of glass close to the bottom of the pot. When the tube has been brought near the side it is held in place momentarily by a fork-like tool made for the purpose, while the stirring-rod is lifted out, withdrawn from the furnace, and laid aside. Then a rod of round iron, one inch in diameter, bent in the form of an inverted U, is dropped down in such a manner that one leg of the U enters the central hole in the tube and the other leg drops over the outside of the pot. The distance between the legs is such that when the hook is in this position the stirring-tube is held close to the pot-wall. One leg of the U should be about 14 inches long and the other 10 when used with a pot whose height from the shoulder to the rim equals 16 inches. The shorter leg is dropped into the tube. The weight of the hook keeps the stirring-tube in a nearly upright position.[1] The stirring-machine is now pushed to one side and the tuille is slightly lifted. By means of a heavy bar with a chisel bit a small hole is driven between the bottom of the pot and the siege (large enough to get the edge of the bar under the pot), a block of iron is laid on the siege as a fulcrum, and the pot is pried loose. The loosening should not be by sudden jolts on the outer end of the bar but by a steadily exerted force. In that way the giving-way of the gluey material which holds the pot will be gradual and will not lead to a sudden agitation of the glass. The tuille is now lifted high, the pot-wagon is run in and the pot

[1] An alternative method is to lift the tube out of the glass altogether. This seems inherently more likely to produce disturbances of a kind that will cause striae, and practical results seem to confirm this inference.

is clasped in its tongs. Counterweights nearly balancing the weight of pot and contents are lifted onto the outer pole of the wagon, the pot is raised quickly but carefully, brought out, and deposited on a tile. The time which elapses from the moment that stirring stops until the pot rests on the tile should seldom exceed five minutes, and when the working force is well organized and matters proceed without a hitch, it will probably not exceed

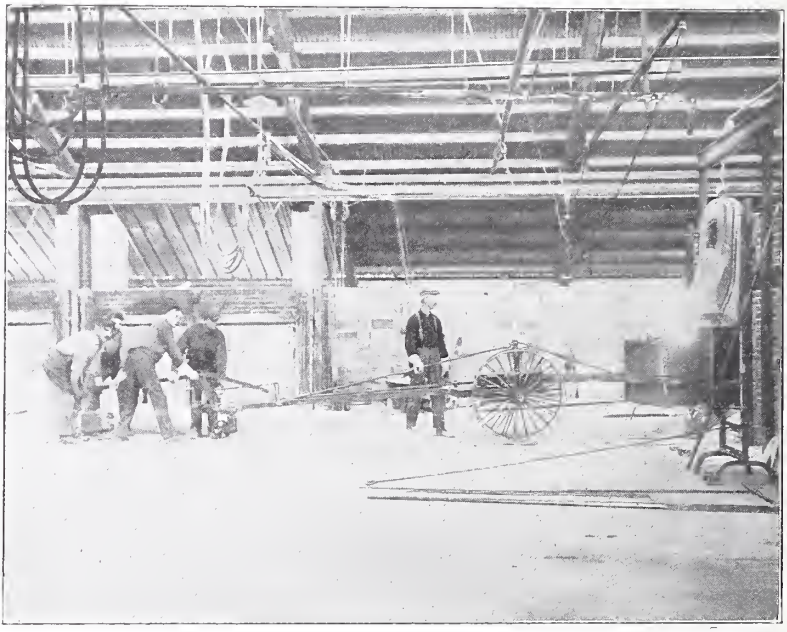


Fig. 3.—Removing pot of glass from the furnace.

three. Fig. 3 illustrates the operation at the moment that the pot is being withdrawn from the furnace.

The pot is left resting on the tile while the siege is got ready for the next run and a new pot is carried from the hot arch and set in the furnace. Then the pot just finished is placed in the arch left vacant. This procedure, however, may be modified. For instance, we found at one time that a certain lot of pots, which were very good in most respects, showed the bad feature of tending to crack while exposed to the cold air, and in one instance a

large section of pot-wall fell out and allowed nearly half the contents of the pot to escape. For these pots we adopted the practice of having an empty arch heated and ready, into which the pot of glass was carried directly from the furnace. The pot to

TABLE 2.
BATCH COMPOSITIONS.

	Medium Flint $n_D = 1.605$ $\nu = 37.6.$	Baryta Flint $n_D = 1.619$ $\nu = 37.6.$	Light Crown $n_D = 1.516$ $\nu = 60.0.$	Boro-silicate Crown $n_D = 1.511$ $\nu = 64.1.$	Light Flint $n_D = 1.580$ $\nu = 41.1.$	Barium Crown $n_D = 1.570$ $\nu = 57.0.$	Dense Flint $n_D = 1.640$ $\nu = 34.6.$
Sand	100	100	100	100	100	100	100
Pb_3O_4	90.50	89.10	....	....	68.06	....	124.10
or	or	or			or		or
PbO	88.40	87.05	...	....	66.46	....	121.20
K_2CO_3 (anhydr.)	10.30	21.30	10.00	15.60	9.44	5.20	5.28
KNO_3	7.81	9.63	12.39	14.00	8.77	11.30	10.30
Na_2CO_3	15.40	....	19.30	18.15	20.51	15.09	16.40
$BaCO_3$	....	19.00	4.69	....	....	75.28	....
ZnO	....	6.95	....	....	....	18.25	....
H_3BO_3	....	....	....	21.00	....	13.60	....
$CaCO_3$	....	....	20.00	5.00	....	....	....
Al_2O_3	....	....	....	....	....	2.07	....
As_2O_3	0.60	0.60	1.00	0.40	0.74	0.61	0.72
Percentage Equivalents.							
SiO_2	47.94	43.80	71.56	70.04	52.72	48.85	41.68
PbO	42.38	38.12	....	....	35.03	....	50.52
K_2O	5.07	8.32	9.01	12.02	5.54	4.30	3.50
Na_2O	4.32	....	8.08	7.44	6.32	4.31	4.00
ZnO	....	3.05	....	....	....	8.91	....
BaO	....	6.47	2.61	....	....	28.57	....
B_2O_3	....	....	....	8.30	....	3.75	....
CaO	....	....	8.02	1.92	....	....	....
Al_2O_3	....	....	....	....	....	1.01	....
As_2O_3	0.29	0.26	0.72	0.28	0.39	0.30	0.30

be used next was heated in another arch. An alternative device for pots which showed a tendency to split open was to pass a chain around them and fasten it.

Under some circumstances it may not be necessary to place the pot in a heated arch to anneal it; instead it is allowed to cool

(with proper protection) on the floor of the furnace-room. Again some glasses may be poured on a casting-table and rolled like plate glass. These matters will be discussed in other papers.

Batch Compositions for Various Types of Glass.

The tables accompanying the article by Zschimmer, previously referred to, contain much valuable information on the compositions of many optical glasses. From these alone it is possible to reproduce almost any optical glass that is made, or we may use them to produce modifications of given types by interpolating between two or more adjacent types. In connection with the work on optical glass that the Geophysical Laboratory has done a great deal of information on the variation of optical properties with composition has been acquired and will soon be published. Thus a quantity of valuable data for producing types of glass of required constants will be available. At the present time the Laboratory is restrained by agreement from publishing any batches which the various plants have themselves developed. This restriction does not apply to batches which the men of the Laboratory introduced, and those which are given herewith in Table 2 fall in this category. Previously given data on temperatures apply to these glasses specifically.

Concluding Remarks.

During the course of this article the writer has taken occasion frequently to emphasize the necessity of attention to details. It may seem that too much stress has been laid upon this and that such matters as following closely the temperature of each pot of glass demand too much skilled labor, and are, after all, unnecessary. This view would be a great mistake. It is surprisingly easy to spoil a pot of glass by a little lack of attention, and the value of one pot will cover a great deal in the matter of wages. In order to get good results in optical glass making it is not possible to conduct operations on a routine that does not take account of variations in conditions which are constantly occurring. One must, instead, treat the process as a laboratory operation carried out on a large scale, and must provide for attention to each pot individually as its requirements demand.

The making of optical glass belongs to a class of operations which American manufacturers have shown a tendency to avoid, apparently preferring to exercise their talents in the direction of turning out great quantities of materials in which each unit may be made to follow the same routine. Products of a more highly specialized kind, such as optical glass, have been obtained from abroad. It seems hardly necessary to emphasize here the lesson which has been taught as to the mistake of a national acceptance of such a policy. It may be well to point out, moreover, that recent events in several branches of industry have demonstrated that there is no lack in this country of men of the necessary scientific and technical equipment to take charge of manufacturing operations of this class and to see that they are properly carried out. It seems that in processes of this kind the same incentives should appeal to manufacturers as in making a good job of any other piece of work in which skill, training, and intelligence are required.

Not all the problems connected with the making of optical glass of a superior quality have been solved. There is still much need of research in order to throw light on certain steps of procedure and remove them from the category of rule-of-thumb operations, and to make more certain the elimination of defects and the production of material that will exactly meet requirements. Some of the subjects for study which may be suggested as promising are a fuller investigation of the function played by arsenic in glass melts and of the amount of volatilization of different ingredients under various conditions, the viscosity of glasses through the temperatures at which stirring is carried on, and the conditions governing the solution of gases in melts. It is to be hoped that some of the manufacturers will turn their attention to having such problems investigated. It is by the inauguration of an enlightened policy of this kind that we in this country may hope to keep in the van in the manufacture of such highly specialized products as optical glass.

Carnegie Institution of Washington,
Geophysical Laboratory,
Washington, D. C.,
February, 1919.

AN IMPROVED METHOD OF OPTICAL GLASS MANUFACTURE.

By George W. Morey.

The usual practice in the manufacture of optical glass consists of the following sequence of furnace operations:[1]

1. **Heating the Pot.**—The pot is carefully pre-heated in a small subsidiary furnace, called a pot arch; from there it is "set" or transferred to the furnace. The pot is usually set at a temperature of about 1050° C, and it must be heated up to the melting temperature of the glass batch, about 1400° for most glasses, before filling. The best practice is to over-burn the pot before commencing the fill.

2. **Filling the Batch.**—The batch, or batch mixed with cullet, is fed into the pot in several installments until the fill is complete. The details of this process differ from plant to plant.

3. **Fining or Planing.**—After the fill is complete the glass is left undisturbed for several hours, primarily to give time for the bubbles to rise to the surface. The temperature during this period is high; in some places it is the practice to use a higher temperature for the fining operation than for the fill.

4. **Intermittent Hand Stir.**—After the fining period is complete it is customary to stir the glass by hand, intermittently; a common schedule is to hand stir for 15 minutes every two hours. This process removes the bubbles of gas adhering to the side and bottom of the pot, and helps to secure uniformity in composition.

5. **Machine Stir.**—After the period of intermittent hand stirring is complete, the glass is stirred continuously, a stirring machine being used. As a rule, soon after putting on the machine, the fire is turned off and the glass allowed to cool, stirring being continued until the pot is removed from the furnace. The

[1] It is assumed that the reader is familiar with standard glass making processes, described in the preceding paper by C. N. Fenner.

operations summarized above take from two to three days in the furnace, the actual time depending on local practice. When, as is usually the case, the empty pots are pre-heated in pot arches, and the finished glass cooled in an appropriate subsidiary apparatus, a melting furnace will yield one pot of glass every two days.

After considerable experience in manufacturing optical glass, certain of the usual operations seemed to be inadequate or illogical, and soon after taking charge of the Optical Glass Plant of the Spencer Lens Company for the War Industries Board, I devised a new schedule, radically different from the above, which may be called the "24-hour" process. Because of its importance in practically doubling the production of optical glass, a basic material in the manufacture of fire control instruments, the details of the process were communicated to the Military Optical Glass and Instrument Section of the War Industries Board, and was communicated by them to the other manufacturers of optical glass.

In the first place, the filling operation required modification. Melting of the batch takes place from the top downward; the upper layer sinters together, then the more easily fusible components trickle down, leaving the upper layers impoverished in the substances usually called "fluxes." This results in the surface becoming high in silica; this was proven by skimmings from both crown and flint batches, which not only showed an actual accumulation of partially dissolved quartz grains, but also had a refractive index lower than that of the rest of the glass.

The fluxes, especially lead, tend to settle to the bottom; this is proven by the dark layer always brought up when hand stirring is begun; by tests made by plunging a long iron rod into the glass and quickly withdrawing it; and, by the examination of partially melted batches which have been removed from the furnace because of leaks, and broken after cooling. In the latter case, the preponderance of silica mentioned above has also been observed.

This initial inhomogeneity, it is true, is removed by the subsequent stirring operation, and this probably accounts for its harmful effects having been overlooked. One of these harmful effects,

especially in flint glasses, is due to the fact that lead-rich mixtures (for example, the extra dense flints) are extremely corrosive on the pots. By the older process this lead-rich layer is allowed to lie on the bottom of the pot throughout the filling and the fining periods, both of which periods are of some hours' duration and of extremely high temperature. This necessarily results in greatly increased pot corrosion. In addition, the impoverishment of the upper layers in fluxes increases the time required for complete solution of the batch ingredients.

The obvious remedy is to stir during the fill.[1] This is not feasible until the pot is a little over half full, because the stirring rod cannot be floated before this, but as soon as feasible, it should be begun, and the melt should be stirred each time new batch is added. Making such a stir during the fill should diminish pot corrosion, give a better quality glass, glass freer from color, striae and stones, and should hasten the solution of the batch and thereby shorten the melting process.

After the fill is completed the glass must be freed from bubbles of gas arising from the decomposition of carbonates and nitrates in the batch, and from the water in the batch ingredients. In some cases these volatile components comprise one-fifth of the weight of batch filled. By the old process the bubbles are mainly removed during the fining operation, the glass being kept hot and undisturbed for some time to allow the bubbles to rise to the surface. The intermittent hand stir that follows, supplements the fining period by removing the layer of bubbles which adheres to the pot-walls, and also tends to secure uniformity in composition throughout the melt.

That seemed an illogical way to secure the desired result. In the first place, common experience is that bubbles in other liquids are more rapidly removed by stirring than by quiescence. It seemed reasonable to suppose that in a pot of glass also the bubbles will be more rapidly removed by stirring. Moreover, in the usual process the glass is not homogeneous in composition

[1] Since presenting this paper I have heard that at one time stirring during the fill was tried at the Charleroi plant of the Pittsburgh Plate Glass Co.; others doubtless have made the same attempt.

until after the fining period; the upper layer is deficient in fluxes, and hence melts less rapidly than the mass of the glass. The margin in composition between an unworkably viscous glass and a workable glass is a narrow one; the stirring should prevent the upper portion being deficient in fluxes and hence too viscous to permit the free passage of bubbles. It seemed a reasonable supposition, therefore, that two of the operations to which optical glass is usually subjected could be dispensed with, namely, the long fining period and the period of intermittent hand stirring. In other words, it seemed that better results could be obtained by putting on the stirring-machine immediately after the fill, and stirring continuously until the glass was free from bubbles. With this modification could well be combined a hand stir during the fill, as mentioned before.

The best test of the above reasoning is the results. The new schedule was tried out on a melt of flint glass having an index of 1.617. The melt was run at the usual temperature for this glass, 1390° C. Fills were made at 2-hour intervals, the first fill being about $^1/_3$ of the total batch, and on each subsequent fill the pot was heaped up with batch. As soon as possible a hand stir was made; ten hours after the first fill the stirring-machine was put on; at this time there was still undissolved batch, not all quartz. The stirring-machine was run at a good speed, both with a circular and a vertical motion. After six (6) hours the glass seemed free from bubbles; the fire was accordingly turned off and the pot cooled and removed as usual. The entire process, from the time the pot was set until the melt was out of the furnace and another pot set, was 24 hours. When the glass was examined it proved to be of the best quality: wholly free from bubbles, of greatly improved color, and also freer from striae than usual.

The details of the process were subsequently modified, and special schedules were worked out for the different types of glass. Following is a sample schedule, being that for a flint, having $n_D = 1.617$, $\nu = 36.5$. The pot used is 26 inches high and 28 inches in diameter, inside dimensions. The time of filling in the cullet is taken as zero hour.

SCHEDULE FOR MF_2 GLASS.

Time: hours.	Run at 1390° C.
0:00	Add cullet
1:00	Fill pot 3/4 full of batch
2:30	Fill pot with batch
4:00	Hand stir; fill pot with batch
5:30	Hand stir; fill pot with batch
7:00	Hand stir; fill pot with batch
7:30	Stirring-machine on
15:00	Gas off.

When cooled to the proper temperature the pot of glass is removed from the furnace and slowly cooled in a pot arch. A new pot is set into the furnace and given a preliminary burning, so that in 24 hours all is ready for another melt.

It may be well to emphasize one point of difference between the longer process and the 24-hour process. In the former the melt, often with an unduly corrosive layer on the bottom, remained in contact with the hot pot for from 20–30 hours from the time the last fill was made until the gas was turned off; in the 24-hour process the corresponding period of maximum corrosive action is 5 or 6 hours. As the greater part of the contamination of glass by iron, with our present raw materials, comes from the pot, the superiority of the newer process is obvious. Better color results from smaller pot contamination, as does also a greater freedom from striae, less trouble from stones, and less pot breakage.

The first experimental pot of glass made by the 24-hour process proved a complete success. Since that time some 350 pots of glass have been made, and all by the shorter schedule. They comprise practically all types of glass; flints, from an extra dense flint with refractive index of 1.76 to an extra light flint with index of 1.55; soft crowns and ordinary crowns of three different types; several types of borosilicate crown; several barium crowns, baryta flint with index 1.56 to a dense baryta flint, index 1.62. Without exception the new process has produced a better glass than the old, with a doubling of production and correspondingly lower cost.

SPENCER LENS GLASS PLANT, HAMBURG, N. Y.
CARNEGIE INSTITUTION OF WASHINGTON,
GEOPHYSICAL LABORATORY, WASHINGTON, D. C.
February 12, 1919.

A COMPARISON OF AMERICAN CHINA CLAYS AS PORCELAIN INGREDIENTS.

By Arthur S. Watts.

As a result of a study of the numerous attempts during the war to make a substitution of American clays not only for foreign clays but also for other American china clays, we are convinced that very few users of American china clays or kaolins are aware of the serious results which may develop from the indiscriminate substitution of one clay for another—even though the sources of supply may be separated by but a few miles and the mode of occurrence be apparently the same. To the average user of china clay, the terms Delaware kaolin, North Carolina kaolin, South Carolina kaolin and Georgia kaolin are ones which condemn or recommend a clay—depending upon the previous experience of the consumer with materials sold under those names. The Florida kaolin has, fortunately established its identity more definitely and most users of white clays are acquainted with its merits and limitations.

The Bureau of Mines will publish, in the near future, data on the clay from each mine being operated in the Atlantic Coast States during 1918, and from these data an opportunity for a careful comparison can be secured.

The samples tested in the present investigation were taken from commercial stock—the selection of the sources of the clays being controlled chiefly through convenience. No attempt was made to obtain the best product from any one district. The clays selected were as follows:

Sample O—M. W. M. English China clay. Moore and Munger, 29 Broadway, New York.

Sample A—Delaware kaolin. Newark China Clay Co., P. O. Box 220, Wilmington, Del. (Shipped from Thompson, Del.)

Sample B—North Carolina kaolin. Hand Clay Co., Canton, N. C. (Shipped from Woodrow, Haywood County, N. C.)

Sample C—North Carolina kaolin. Harris Clay Co., Dillsboro, N. C. (Shipped from Dillsboro, Jackson County, N. C.)

TABLE I

	Bodies.	Per cent water of plasticity.	Per cent drying shrinkage.	Per cent total shrinkage		Per cent absorption.		Per cent warpage.		Per cent translucency.		Color.	
				Cone 7.	Cone 10.	Cone 7.	Cone 10.	Cone 7.	Cone 10.	Cone 7.	Cone 10.	Cone 7.	Cone 10
O.	Eng. china clay body	26.0	5.0	13.4	16.7	1.4	0.0	0.14	0.37	7	1	1	2
A.	Del. kaolin body	25.8	4.0	11.0	13.8	1.9	0.0	0.25	0.32	6	2	9	5
B.	N. C. kaolin body	26.1	3.2	10.0	11.7	4.0	0.2	0.16	0.18	8	6	8	4
C.	N. C. kaolin body	19.2	2.8	10.0	12.0	2.7	0.0	0.27	0.37	3	3	3	1
D.	S. C. kaolin body	23.4	2.8	11.6	11.8	3.6	0.3	0.23	0.29	4	7	7	7
E.	S. C. kaolin body	25.8	2.0	12.0	12.0	2.7	0.0	0.30	0.33	1	5	6	9
F.	S. C. kaolin body	28.8	2.0	12.2	12.7	3.5	0.2	0.17	0.18	5	4	5	3
G.	Ga. kaolin body	23.9	3.6	12.2	13.4	4.1	0.4	0.19	0.40	2	8	4	8
H.	Ga. kaolin body	24.1	3.1	9.0	11.5	4.0	0.0	0.19	0.15	9	9	2	10
I.	Ga. kaolin body	24.8	3.5	10.6	13.5	4.4	0.7	0.15	0.14	10	10	10	6

Sample D—South Carolina kaolin. Interstate Clay Co., Sumter, S. C. (Shipped from James Crossing, Sumter County, S. C.)

Sample E—South Carolina kaolin. McNamee Kaolin Co., Cumberland Bldg., Broadway at 54th St., New York. (Shipped from Bath, Aiken County, S. C.)

Sample F—South Carolina kaolin. Edisto Kaolin Co., Bridgeport Nat'l Bank Bldg., Bridgeport, Conn. (Shipped from Samaria, Aiken County, S. C.)

Sample G—Georgia kaolin. Edgar Bros. Co., Metuchen, N. J. (Shipped from McIntyre, Wilkinson County, Ga.)

Sample H—Georgia kaolin. Georgia Kaolin Co., Macon, Ga. (Shipped from Dry Branch, Twiggs County, Ga.)

Sample I—Georgia kaolin. Golding Sons Co., Butler, Ga. (Shipped from Butler, Taylor County, Ga.)

A body having the following composition was chosen for testing the properties of each clay:

	Per cent.
Feldspar (Canadian)	20
Whiting	2
Tenn. ball clay No. 7	10
Florida kaolin (Edgar Plastic Kaolin Co.)	10
Clay being tested	25
Flint	33

These bodies were wet-ground for four hours in ball mills, transferred to plaster absorption bowls lined with cambric to prevent contamination, and were then thoroughly wedged and molded into the following shapes:

Wedges, 3″ long, 1″ wide, tapering from $^3/_4$″ to 0″.

Warpage trials, 7″ long, 1″ wide, $^1/_2$″ thick.

Cups, approximately $3^1/_2$″ diameter by 2″ high (jiggered).

The warpage trials were placed on wedges in firing, with 5″ span.

Trials from each body were fired in two burns to cones 7 and 10, the data secured from the burned pieces being given in the table on the preceding page.

Conclusions.

A study of the foregoing data indicates that the variation in the physical properties of the clays tested is worthy of serious consideration. The water content for the best working plasticity is fairly constant, 23.4 per cent to 26.1 per cent, except in the

cases of the Harris North Carolina kaolin which required only 19.2 per cent and the Edisto South Carolina kaolin which required 28.8 per cent water.

The drying shrinkage was greatest in the body containing English china clay, followed by the Delaware, Georgia, North Carolina and South Carolina kaolins, respectively.

The American clay bodies, when fired to cone 7 and 10, had a lower total shrinkage than the English clay body in every case.

The rates of vitrification of the different clays, as evidenced by the absorptions of the different bodies, were somewhat surprising. The bodies containing Georgia kaolin were more porous than any of the others when fired to cone 7, with the South Carolina kaolin bodies next, followed by the North Carolina, the Delaware, and the English china clay body, which was the most vitreous at cone 7. The same order of porosity was also noted in the pieces fired to cone 10.

The warpage behavior of the bodies is interesting, since the English china clay body, although the most vitreous at cone 7, shows the least warpage when fired to this cone. At cone 10, however, it had about the same warpage as the other bodies, there being only three exceptions, B, the Hand North Carolina kaolin, F, the Edisto, South Carolina kaolin and H, and I, the Georgia Kaolin Co. bodies which had exceptionally low warpage at cone 10.

Probably the most important consideration in studying these bodies are the translucency and color. It will be noted that the bodies showing the highest translucency at cone 7 do not have the highest translucency at cone 10. In but one case does the relative translucency of a body remain the same at both cones. We judge from this that the temperature at which a body is to be fired is of vital importance in the selection of the kaolin to be used. At cone 10, which is about the average maturing cone for whiteware bodies, the English china clay showed the best translucency, then followed the Delaware kaolin, a North Carolina kaolin, a South Carolina kaolin and lastly the Georgia kaolins.

In considering the color, which is of even more importance than translucency, the results at cone 10 indicate the following order of excellence: North Carolina, English, South Carolina,

North Carolina, Delaware, Georgia, South Carolina, Georgia, South Carolina, and Georgia kaolins. The difference in color, although small, is worthy of careful consideration and, while the other properties remain reasonably constant for a limited area, the color and translucency vary widely within limited areas and only by careful test and selection can the best results be obtained. The color classification as given in the table is the result of grading by only two persons, and in the absence of any standard method of test is, of course, open to the fault of the personal factor.

Department of Ceramic Engineering,
Ohio State University,
Columbus, Ohio.

AMERICAN CERAMIC SOCIETY.

Acquisition of New Members during February, 1919.

Associate.

Ahrens, Robt. S., California Metal Enam. Co., Los Angeles, Cal.
Agge, Franklin, Republic Metalware Co., Buffalo, N. Y.
Arensberg, C. C., McCullough-Dalzell Crucible Co., Pittsburgh, Pa.
Barnes, T. R., Barnes Mfg. Co., Mansfield, Ohio.
Blair, J. A., Kushequa Brick Co., Kushequa, Pa.
Buckley, J. L., Phila. Drying Machine Co., Philadelphia, Pa.
Chaffin, Edwin G., Norton Company, Worcester, Mass.
Collin, Louis, Alfred, N. Y.
Coxon, J. B., Standard Sanitary Mfg. Co., Kokomo, Ind.
Crawford, George E., Alfred, N. Y.
Crume, Wm. H., Crume Brick Co., Dayton, Ohio.
Czerny, Leonard, Mosaic Tile Co., Zanesville, Ohio.
Dains, I. F., Western Stoneware Co., Monmouth, Ill.
DeWitt, Bert G., Belmont S. & E. Co., New Philadelphia, Ohio.
Dimcombe, Geo. H., Jr., Evens & Howard Fire Brick Co., St. Louis, Mo.
Edwards, W. L., Wheeling Sanitary Mfg. Co., Tiltonville, Ohio.
Enright, Bernard, Allentown, Pa.
Fisher, E. E., Federal Glass Co., Columbus, Ohio.
Foskett, John J., Foskett Co., Inc., Port Jervis, N. Y.
Foster, Harry D., Mosaic Tile Co., Zanesville, Ohio.
Goodwin, Herbert, Salem China Co., East Liverpool, Ohio.
Hagar, Donald, Alfred, N. Y.
Harvey, Ives L., Bellefonte, Pa.
Henshaw, S. B., Libbey-Owens Glass Co., Charleston, W. Va.
Heuisler, Philip I., Maryland Glass Corp., Baltimore, Md.
Hinrichs, Carl G., Hinrichs Laboratories, St. Louis, Mo.
Hostetter, J. C., Geophysical Laboratory, Washington, D. C.
Hunt, Marsden H., Univ. of Illinois, Urbana, Illinois.
Jenks, H. P., Hook Patent Kiln Co., Columbus, Ohio.
Jewett, Richmond, Jewett Refrigerator Co., Lackawanna, N. Y.
Kirk, Herbert S., Universal Sanitary Mfg. Co., New Castle, Pa.
Lapp, G. W., Lapp Insulator Co., LeRoy, N. Y.
Limberg, Frank, Limberg Enameling Works, Cincinnati, Ohio.
Lin, S. Y., Ohio State University, Columbus, Ohio.
Lindsay, G. W., Denver Fire Clay Co., Denver, Ohio.
Mahoney, W. T., Cahill Iron Works, Chattanooga, Tenn.
Memory, N. H., Isbell-Porter Co., Newark, N. J.

Palmer, Chas. S., Mellon Institute, Pittsburgh, Pa.
Porter, J. Edward, Box 785, Syracuse, N. Y.
Rentschler, M. J., J. H. R. Products Co., Willoughby, Ohio.
Roddewig, Ed., Iowa State College, Ames, Iowa.
Sauereisen, C. Fred, Empire China Works, Brooklyn, N. Y.
Schurtz, D. D., Frink Laboratories, Lancaster, Ohio.
Shively, R. R., Monongah Glass Co., Fairmont, W. Va.
Smoot, C. E., 1900 Santa Fé Ave., Los Angeles, Cal.
Sosman, Robert B., Geophysical Laboratory, Washington, D. C.
Stowe, G. T., Lock Haven Fire Brick Co., Lock Haven, Pa.
Vollrath, W. J., Porcelain Enameling Association, Sheboygan, Wis.
Wainford, Richard H., Trenton Flint & Spar Co., Trenton, N. J.
Williams, R. G., Stanley Insulating Co., Great Barrington, Mass.
Williams, W. S., Bureau of Standards, Pittsburgh, Pa.

Foreign.

Cole, L. Heber, Mines Branch, Dept. of Mines, Ottawa, Ont., Canada.
Scott, Alex., Central School of Science & Technology, Stoke-on-Trent, Eng.
Skidmore, George R. J., Geo. Davidson & Co. Glass Works, Gateshead-on-Tyne, England.

Contributing.

Foote Mineral Co., Inc., Philadelphia, Pa.
Lindsay Light Co., Chicago, Ill.
Monongah Glass Co., Fairmont, W. Va.
Stanley Insulating Co., Great Barrington, Mass.
U. S. Glass Co., Pittsburgh, Pa.
L. J. Houze Convex Glass Co., Point Marion, Pa.
Macbeth-Evans Glass Co., Pittsburgh, Pa.
Phoenix Glass Co., Pittsburgh, Pa.
Trenton Flint & Spar Co., Trenton, N. J.
Western Stoneware Co., Monmouth, Ill.

LOCAL SECTIONS.

Chicago Section.

The fifth meeting of the Chicago Section of the American Ceramic Society was held at the City Club, Chicago, January 11, 1919. In the absence of President W. D. Gates, Vice-President I. E. Hardy presided. The minutes of the previous meeting were dispensed with and the election of officers for the ensuing year was taken up with the following result:

Chairman, I. E. Hardy, Tiffany Enameled Brick Co., Momence, Ill.

Vice-Chairman, A. F. Hottinger, Northwestern Terra Cotta Co., 2525 Claybourn Ave., Chicago, Ill.

Secretary and Treasurer, H. T. Bellamy, Hawthorne Works, Western Electric Co., Inc., Chicago, Ill.

Councilor, C. W. Parmelee, University of Illinois, Urbana, Ill.

Chairman of Program Committee, R. R. Danielson, Benjamin Electric Manufacturing Co., Des Plaines, Ill.

Chairman of Membership Committee, B. S. Radcliffe, Midland Terra Cotta Co., Lumber Exchange, Chicago, Ill.

After the election of officers, the three following very interesting papers were presented and discussed:

"*Production*," G. G. Lawson, Northwestern Terra Cotta Co., 2525 Claybourn Ave., Chicago, Ill.

"*The Use of Powdered Fuel*," F. L. Steinhoff, "Brick and Clay Record," 610 Federal St., Chicago.

"*Clay Mines in Tennessee and Kentucky*," Prof. C. W. Parmelee, University of Illinois, Urbana, Ill.

Adjourned.

H. T. Bellamy,
Secretary.

ACTIVITIES OF THE SOCIETY

Proceedings of the Board of Trustees at Pittsburgh, February 2-5, 1919.

A meeting was held in the Secretary's room at the Fort Pitt Hotel on Sunday evening, February 2nd, at 7.30. There were present, Homer F. Staley, President, A. F. Greaves-Walker, Vice-President, R. K. Hursh, Treasurer, E. T. Montgomery, R. D. Landrum, G. H. Brown, Trustees, Chas. F. Binns, Secretary, and Norah W. Binns, Assistant Secretary.

The Secretary read a draft of the report of the Board to be presented to the Society. After discussion and some minor alterations this was by vote adopted.

It was voted that the proceedings of the Board during the year, including motions and resolutions passed, be published in the Year Book together with the dates at which they were made, and that current motions in their final and complete form be transmitted to the Editor monthly by the Secretary for publication in the Journal.

It was voted to complete thirty sets, if possible, of the Transactions, bound in cloth.

It was voted that no ballots for the election of incoming officers be received later than 10 A.M. on Tuesday, February 4th.

In response to an inquiry by the Secretary as to the date upon which membership should be considered to have lapsed, it was voted to send the Journal for January, February and March to all members irrespective of payment of dues.

It was voted that in the opinion of the Board the Society should continue to be represented at the Exposition of Chemical Industries.

Letters were read which had passed between Lt.-Col. Orton and Dr. J. W. Mellor of the English Ceramic Society with reference to a possible joint meeting of the two Societies in this country in the summer of 1920. It was voted that the Secretary issue a formal invitation to the English Ceramic Society to attend a joint meeting at that time, and that a committee of three, with power to add to their number, be appointed to make the necessary arrangements.

A request was presented from Mr. F. H. Rhead for the formation of a Professional Division on Ceramic Decorative Processes. It was voted that the Secretary advise Mr. Rhead that if ten members apply for the organization of such a Division an authorization will be given.

Several modification of the Rules were suggested and it was voted that these be referred to the Committee on Rules for consideration and, if approved, for presentation to the Society before the close of the Convention.

A list of names which had been presented for elevation to Active Membership was passed in review and some tentative changes were made.

The Board met at the same place on Monday evening, February 3rd, at 9 o'clock. All the members were present, together with Mr. R. H. Minton, Vice-President elect, and Mr. M. F. Beecher, Trustee elect.

The names of 102 resident Associates and 13 foreign Associates were passed for election to Active Membership.

It was voted to discontinue the employment of a stenographer to report discussions and to engage a local stenographer as occasion might arise for the report of the business meeting of the Society.

It was voted that the Secretary might at his discretion consider, after a reasonable lapse of time, that a motion before the Board in correspondence is decided if a minimum of three votes shall have been received and recorded.

It was voted that the Treasurer furnish a satisfactory bond in the sum of $5000 and that the Secretary's bond of $2000 be renewed.

It was voted to remove the names of all German members from the rolls, on the ground that their dues have not been paid.

A meeting of the newly-elected Board was held in the Assembly Room on February 5th at 6 o'clock. All the members were present.

It was voted that a Professional Division on Wall and Floor Tile be authorized and that the President appoint a chairman.

It was voted that the resolution which empowered the Secretary to purchase copies of Volume XIX at $5 each be amended to read "at the market price."

It was voted that one copy of the "Directory of Dealers in Raw Ceramic Materials" be sent to each Member and Associate of the Society, except to those who have already purchased them, and that the money paid for such purchases be refunded.

JOURNAL
OF THE
AMERICAN CERAMIC SOCIETY

A monthly journal devoted to the arts and sciences related to the silicate industries.

Vol. 2 **March, 1919** **No. 3**

EDITORIALS.

NATIONAL RESEARCH COUNCIL.

Pursuant to the request of the President of the National Academy of Sciences, that the American Ceramic Society nominate one representative for appointment as a member of the National Research Council in the Division of Chemistry and Chemical Technology, President Stull has recently nominated Mr. A. V. Bleininger to represent the Society in this Division and the formal appointment has been made by the President of the Academy.

In accordance with an executive order of the President of the United States, dated May 11, 1918, requesting the perpetuation of the National Research Council, a permanent organization for the Council has recently been adopted. In order to secure an effective federation of the chief research agencies of the country, it was decided that a majority of the members of each Division of the Council should consist of representatives of the leading national scientific and technical societies in the United States. On the recommendation of the Executive Committee of the former Chemistry Division, the new Division of Chemistry and Chemical Technology is to be initially constituted of representatives from the American Chemical Society, American Electrochemical Society, American Institute of Chemical Engineers, American Ceramic Society and Divisional nominations.

The initial meeting of the new Division was held in Washington on March 21st and 22nd. The salient features of the work accomplished at this meeting were the consideration of the establishment of research and industrial fellowships; a discussion of the formation of an International Chemical Society and the

promotion of international coöperation in publication; and a consideration of the patent situation.

At a meeting of the Executive Committee of the Division of Chemistry and Chemical Technology the following resolutions concerning the formation of a Committee on Ceramic Research were adopted:

"1. That the Division invite the American Ceramic Society to form with it a joint committee on ceramic research of which the representative of the Ceramic Society on the Division shall be ex-officio chairman.

"2. That the Committee consist of five members, two to be nominated by the executive committee of the Division and two by the Board of Trustees of the American Ceramic Society.

"3. That this committee undertake the following work:

"(*a*) Outline a number of important research projects of a character which can be undertaken in university laboratories.

"(*b*) Ascertain in the case of each project what institution is able and willing to undertake the investigation.

"(*c*) Raise by subscription from the industries a Fellowship Fund for each project, this fund to be used for the establishment of a two-year Fellowship, a portion of the fund to be available for traveling expenses, apparatus, supplies, etc. It shall be understood that all funds raised for such purpose shall be paid to the treasurer of the National Research Council, to be disbursed by him under the direction of the Committee on Ceramic Research; and that in connection with all such Fellowships regular reports shall be rendered to the chairman of the Committee and that a copy of the report be transmitted to each subscriber to the fund.

"(*d*) Make a study of the desirability and possibility of compiling a research census of the ceramic research in progress in the country, and, if the preparation of such an index is deemed desirable and practicable, undertake its compilation.

"(*e*) In coöperation with the Section on Industrial Research of the National Research Council, endeavor to secure the establishment of coöperative research laboratories in those branches of ceramics where such an undertaking seems feasible.

"(*f*) Secure if possible a statement of the plans of the Director of the Census with reference to collecting ceramic statistics in the coming census, and endeavor to secure such notification of, and additions to, these plans as it thinks desirable in order that such statistics shall be as complete and as useful as possible.

"(*g*) Take the initiative in attempting to secure the inauguration of cooperative effort by the appropriate agencies in the country in carrying out certain broad projects in ceramic research, such, for example, as the Cooperative Survey of Ceramic Raw Materials, initiated at the Pittsburgh Conference last May."

Upon the recommendation of President Stull, Mr. Homer F. Staley and Dr. Robert B. Sosman have been appointed by the Board of Trustees as the representatives of the American Ceramic Society on the Committee on Ceramic Research. The Society is to be congratulated in being so ably represented in the work of the Division by Messrs. Bleininger, Staley and Sosman. It is to be hoped that each member of the Society will lend his assistance to these men in the coördination and advancement of ceramic research in accordance with the comprehensive plan outlined.

NATIONAL FIRE PROTECTION.

The considerations which made it appear desirable for the American Ceramic Society to become a member of the National Fire Protection Association were purely those of service, first to the country at large, by supporting and assisting in the important work of keeping down our enormous fire losses, and second, to that portion of the clay-working industry which produces building materials of various sorts.

The latter service should be two-fold, direct and indirect. The National Fire Protection Association establishes standards for many things pertaining to fire prevention and protection, including fire resistive construction. It would appear that the American Ceramic Society should be able to render a direct service to the industry producing structural clay products, by taking part in the drafting of structural standards. As to the indirect service, the support and assistance which the Society could give to the Fire Protection Association would be of benefit to the clay industry in common with all industries which produce fire resistive building materials.

It would appear that more active coöperation with the N. F. P. A. would strengthen the Society with certain branches of the clay industry which are now not as well represented in the Society as is desirable and that in the end this activity would be of benefit in a number of ways.

ADDRESS OF THE RETIRING PRESIDENT.[1]

HOMER F. STALEY, Washington, D. C.

This is a significant moment in the life of the American Ceramic Society. It marks the opening of the twentieth anniversary meeting. Twenty years is a long time in the life of a society. In that time this Society has grown from a membership of a few score to an enrollment of over twelve hundred. Its publications have increased in amount until now each monthly issue of the Journal contains more material than appeared in the first annual volume of the Transactions. From a little known and less understood organization, the very name of which had to be explained even to clay products manufacturers, it has come to be the greatest technical organization in the world devoted to the advancement of the silicate and allied industries; an organization that is a power in these industries and capable even of giving material assistance to our country in winning a great war.

Custom has established that it is the special privilege, if not duty, of the president of a technical organization to give, on retirement from office, a review of the activities of the society which has honored him and to make such recommendations in regard to its future activities as seem to be desirable. It seems especially fitting that, at the close of twenty years of activity of our organization, such an address should be given.

It would be very easy and pleasant to spend an hour reviewing the many achievements of the Society during the past twenty years. It would be possible to dwell with profit on the activities of the Society during the closing year, which has been the most notable in many ways of any in the history of the organization. The Society has accomplished many things of which it can justly be proud. At present it is apparently in a very prosperous condition, both from the standpoints of membership and finances.

[1] Twentieth Annual Meeting, American Ceramic Society, Pittsburgh, Pa., February 3, 1919.

However, it should be realized by everyone that much of the phenomenal growth of the Society in membership and financial support during the past two years has been based on our promises to make the work of the Society of great practical value to its members. Many things have been done during the past year in fulfillment of these promises and to a large extent the Society is meeting the expectations of its members and friends. Other things remain to be done before the Society gives the full measure of service toward which we are confidently working. It is of these that I wish to speak.

A society like an individual grows both in form and in spirit. Because I believe that the development of form is secondary to development of spirit, and in truth, that the growth of the proper spirit will inevitably result in a workable form of organization, I will state a few recommendations for changes in the form of our Society and then emphasize the vital necessity for proper spirit in the conduct of the Society's affairs.

The changes in Society organizations and extension of activities I desire to suggest are:

(1) The enlargement of the scope of the Journal.

(2) The employment of a full-time secretary-editor.

(3) The establishment of permanent headquarters for the Society.

(4) Increase in the number of voting members.

(5) Definite decision as to the status of the cement and mortar industries in the Society's activities.

That the Journal should be enlarged in scope is well understood by everyone who has given any thought to the matter. The publication of the Journal is a decided improvement over the former practice of issuing only an annual volume of Transactions each year. It enables us to publish quickly the papers read at the annual and other meetings; attracts to our publication some papers that we would not receive under the old plan, and aids in keeping the membership in general in touch with the activities of the Officers, Board of Trustees, and various committees of the Society. The Committee on Publications and the Editor deserve great credit for the remarkably successful manner in which they have handled the launching of the Journal. The

starting of a new technical publication would have been a difficult problem at any time and was especially difficult at a time when the country was in the midst of a great war.

The Journal is not yet giving all the service of which it is capable and which is planned by the Committee on Publications. In order to hold and increase our membership, which means holding and increasing our advertisers, it is essential that each number of the Journal shall contain original papers or other material of practical value to each of the industries represented by our membership. It is hoped that the full development of the system of establishing Local Sections and Professional Divisions will result in the contribution of more papers, especially of the class designated as practical since they deal directly with factory problems. However, the Board of Trustees and Committee on Publications have been considering the advisability of establishing also a review department in the Journal with at least as many subdivisions as there are Professional Divisions in the Society. The plan proposed is not to attempt to duplicate the work of the existing abstract journals carrying ceramic departments by giving meager reports of everything published related to the ceramic industries, but to give rather full reviews of articles of real merit. To conduct such a department properly will cost some money, but the money spent in this way will undoubtedly be a good investment resulting in a larger list of members.

Another improvement in the Journal which has gradually been taking shape is the establishment of a fixed editorial policy. There are certain ideals toward which the Society is striving at all times, certain objects toward which it works at particular times. In the first class may be mentioned promotion of research and technologic development, the establishment of a high standard of professional ethics, and altruistic service to the ceramic industries in various ways. Among the aims of special efforts may be classed such movements as those for increase of membership, for contribution of papers to the Journal, or a large attendance at a meeting. The experience of the past two years has shown that in a society as large as this has grown to be, the new members can not be made enthusiastic for these ideals, and the membership as a whole can not be moved to action

in these special movements by an occasional letter from an officer or the chairman of a committee. It is coming to be the function of the editorial department of the Journal to place these questions before the membership in a systematic manner.

The employment of a full-time secretary-editor has been contemplated by the Society for several years. The lack of sufficient funds has been the deterring influence. We are glad to state that this obstacle has been overcome and funds for this purpose will be available from now on. The employment of a high-grade man to oversee the business affairs of the Society and to act as managing editor of the Journal is necessary to the future success of the Journal and of the other activities of the Society. Many of the Society's activities can be conducted largely through the agencies of the Board of Trustees, Sections, Divisions and Committees; but in addition to these we should have one man whose sole business in life shall be to attend to the affairs of the American Ceramic Society. The Secretary will be kept very busy indeed maintaining a voluminous correspondence, in overseeing the business affairs of the Society, in editing the Journal as to substance and general form, in writing editorials and in securing advertisements. The Society has few men who can fill the position properly and probably most of these will not care to consider a position so insecure of tenure and exposing the occupant to so much criticism. The best man available should be secured; and then the members of the Society should remember that while he will be the servant of the majority of the membership, he will in no sense be subject to the criticisms or wishes of the individual members. In order that the position may be removed from the field of petty politics and that the occupant may be given some assurance of continuity of employment as long as his work is considerd efficient by those in a position to know, it is desirable that the full-time secretary shall be hired by the Board of Trustees, rather than elected by popular vote. This is customary in other societies and can be arranged for by a simple change in the Rules.

The establishment of permanent headquarters for the Society is another corollary of the hiring of a full-time secretary. During the past year the Society seems to have been scattered all over

the country. Some of the Society's mail has gone to the printer, some to the Editor, some to the Secretary and some to the President. Other technical societies have found it advantageous to establish permanent headquarters in some large city, and no doubt the prestige of our organization and the effectiveness of its work will be increased when we are able to do the same.

The proportion of voting members in the Society's list of members should be materially increased. A start has been made in this direction by the alteration of the Rules so that presentation of papers is no longer the sole basis of elevation to membership. Nominations for elevation to active membership in accord with the new regulations have not been as numerous as the Board of Trustees has wished. It is hoped that a large number of nominations will be made during the course of this meeting.

It has been suggested that the grade of Associate Membership be abolished and that all members now in that grade be made Active Members. From the standpoints of democracy and equality of voice in Society affairs, this change would be admirable. However, several grave objections to this procedure should be taken into consideration. It may seriously interfere with the acquisition of members if we raise either the initiation fee or annual dues of new members. There is no need to dwell on this. On the other hand, we should not reduce the income from active memberships. From the standpoints of service to the greatest possible number of people in the ceramic industries and of increasing our possible revenue from advertising in the Journal, it is a good policy for us to carry a certain percentage of members at a loss as far as direct income is concerned. We are now doing this with all associate members of more than two years' standing. For several years the expenditures of the Society have averaged close to eight dollars per member. During the past year it was ten dollars per member. In the interest of good business policy and of fairness to our contributing members, the type of membership that does not pay its way should be reduced to a minimum rather than voluntarily increased.

In the interest of continuity of Society policy it is advisable that members serve a probationary period before being given the voting power. This is a certain kind of Society with certain

ideals. Whether or not it is the best possible type of organization or its ideals are the most laudable, the fact remains that the Society has grown and prospered under this system, and we are reasonably certain that it will continue to prosper if these policies are continued. We know how our present Active members will vote on any proposition involving departure from the fixed ideals of the American Ceramic Society. We know that after a man has been an Associate member of the Society for a few years and has assimilated the spirit of the Society, he too will strive for these same ideals.

The present Rules are really quite elastic on the subject of elevation of Associate members to Active membership. The only requirements are that "Active members must be persons competent to fill responsible positions in the silicate industries," and that they shall be nominated in a certain way and approved by the Board of Trustees. Under these rules if it is the wish of the Society the Board can approve the elevation of any member holding a responsible position in ceramic work as soon as it is satisfied that he is interested in the work of the Society and is sufficiently conversant with the spirit and aims of the organization to be intrusted with the voting power. It should be realized that it is rather difficult for any body of men to decide in a manner satisfactory to everybody concerned who are and who are not worthy of elevation to active membership. Another weak point in the operation of the present Rules is that nominations are required and the present active members will not take the trouble to make nominations. The Board of Trustees should be empowered to act in this matter on their own initiative if they so desire. This is usual in other large technical societies.

The above methods of operating the Rules for elevation to Active membership will augment the income of the Society very materially by placing the bulk of our memberships on a self-supporting basis; will enable the Society to honor new associate members rendering special service to the Society or the silicate industries; and will confer the voting power on all qualified to exercise the privilege. It has the additional advantage that new members can enter the Society at a comparatively low rate; and, since acceptance of Active membership is not compulsory,

they can continue at this rate if they are not interested in becoming Active Members.

If the Society wishes to have Associate members elevated to Active membership on the basis stated above, and there is serious question of our ability to do this under the present Rules, these should be amended. If this is done I would suggest that at the same time the initiation fee for Associate Membership be abolished. Otherwise we will be charging a member two initiation fees in a short time. The abolition of the first fee will greatly facilitate the process of getting new men interested in the work of the Society and thus increase our income from advertising and ultimately from active memberships.

As stated above the alternative plan of making all individual members active members has much to recommend it. May I suggest that if this plan is adopted the initiation fee be abolished and the annual dues be raised to ten dollars? The total income from each member would be about the same under the two plans.

The American Ceramic Society has always claimed that the cement and mortar industries were allied to the distinctively ceramic ones. These industries have refused to recognize this claim, largely because in our literature we have attempted to base the claim on similarity of raw materials. The true kinship is one of technique and technology in that the clay products, glass, enamel, artificial abrasive, cement and mortar industries all make products from non-metallic raw materials in a large way by the use of fire. They use the same general type of equipment and have to deal with the same pyro-chemical and pyro-physical processes. They are allied industries in the same degree that the various metallurgical industries are allied.

The lime manufacturers have recently admitted this common interest and the National Lime Manufacturers Association has designated the Journal of the American Ceramic Society as the official organ for the publication of the technical papers of that Association. We are in a position, therefore, to obtain some memberships and subscriptions from the 1300 members of the National Lime Association. This will result in the formation of a Lime Division in our Society and the publication of considerable material on the technology of lime in the Journal.

To the best of my knowledge the claim of similarity of technique has never been officially presented to the associations of cement and gypsum manufacturers. They both persist in believing that their industries are more closely allied to the purely chemical industries than to the ceramic group. Therefore, although we have received a number of contributions dealing with the technology of cement and gypsum products' manufacture, for which we thank the authors, we have never received any appreciable recognition or financial support from these industries. It is not fair to the industries that are supporting the Society for us to continue to publish material in the interest of industries that give us no support. A decision should be made. If it is possible to gain the support of cement and gypsum industries, an energetic effort to do so should be made. If it is not, we should cease to publish material relating to these industries and spend our money and energy in more receptive fields.

In the past, there has been some antagonism between cement manufacturers and manufacturers of crude clay products, on account of these products being sold for the same uses. This antagonism is dying out as the relative merits and suitability for specific uses of the various products is becoming better known. In the near future we may hope to see cement products and burned clay products sold as complementary materials rather than as antagonistic ones. However, the American Ceramic Society is interested in the making only of cement and burned clay products and not in trade rivalries. No trace of this rivalry should be allowed to appear in our publications or discussions.

The Society has some of the external form of a large society, and is rapidly developing the spirit of one. The form of an organization is merely an incidental shell: the spirit animating the organization is the essential substance that determines whether or no it shall be truly great and effective in its work. From the very beginning, the American Ceramic Society has been animated by high ideals and in many ways by an admirable spirit. The spirit of mutual helpfulness of member to member in solving technical problems has made membership in the Society a joy and an inspiration. The altruistic service to the ceramic industries so ably rendered in many ways in the

Society's activities is a fine example of the successful following of a high ideal. I would not change in the least the spirit of the Society in these respects. They are elements of greatness that make us all proud to be members of the American Ceramic Society.

A spirit of great courtesy and consideration for the feelings and wishes of the individual members of the Society has marked the conduct of the Society's business. This is a beautiful spirit and was very admirable in the early days of the Society, but it is not one in which a great and complex organization, such as the American Ceramic Society is coming to be, can be operated. The inevitable result of this spirit has been that a few members have come to feel that the Rules should not be adhered to strictly, that they should be ignored in some cases and special interpretations put upon them in others.

The Society is now so large and its organization is so complex that the only way its business can be conducted efficiently and without friction is by strict adherence to the Rules. These are the statements we have published of the methods in which the Society's business shall be conducted. As long as it is conducted according to these statements, no one has the right to claim lack of consideration. To permit one member to break the Rules is not an example of courtesy to him, but of injustice to a thousand other members.

In place of the spirit of consideration for individual members, there is developing a desire for orderly conduct of the Society's business and a spirit that puts the vote of the majority above individual wishes. This is the spirit of true democracy, the spirit that made Rome the ruler of the world, and the spirit that animates every great and successful technical organization in existence today. It is the privilege of the Society to alter the Rules; but as long as a rule stands on our books, every member should conform to it whether he personally thinks it is wise and just or not. The will of the Society must take precedence of the wish of the individual.

In one other respect the spirit of the Society should be changed. This is essentially a coöperative organization for the reading and publication of original papers. The fact is that the great majority of our members do not coöperate; they do not present

papers. Only about ten per cent of our membership contribute papers in any one year. About half of these are regular contributors. Five per cent of the membership of the Society has done seventy per cent of the work that has made the Society possible. This is deplorable. Every member of the Society should feel it his duty to present a paper or a short note on some subject at least every other year. Every man in the ceramic industry has opportunity to collect sufficient data to do this. There are very few who lack the ability to present such data in plain-straight-forward English. The real explanation for the dearth of papers is the refusal of the majority of our members to make the effort necessary to write up the data in their possession. They seem oblivious to the fact that the men who do prepare papers are spending time and labor for their benefit. Of course, all members pay dues, but these do not pay for papers—in the case of Associate members they do not even pay for the paper and printing of the Journal.

If this Society is to live and grow and expand, it must have an abundant supply of original papers on ceramic subjects. No plan of reorganization, no activities of committees, no growth in membership, will compensate for the lack of these. All the other activities of the Society are subordinate to and complementary to the one vital function of the publication of ceramic literature. It is probably hopeless to expect that the ideal of every member a contributor will ever be attained, but this is the point toward which we should strive. Every member should realize that he is not doing his full duty to the Society and to the ceramic industries if he does not make the contributions of which he is capable, even at the expense of considerable effort.

In closing, gentlemen, I wish to assure you that I believe the Society is progressing rapidly, that it is making real progress toward becoming a great Society both in form and spirit, and that I firmly believe that in the next few years we will see a remarkable expansion in the membership, activities, and influence of the American Ceramic Society. Permit me to thank you also for the honor you have conferred on me, and to thank you all, especially the other officers, members of the Board of Trustees, and committees for your efficient coöperation in the work of

the Society during the year. To be president of a large technical organization, such as this has come to be, is an interesting psychological experience. To me it has meant an insight into human nature that could probably not have been gained in any other way. It has meant lots of work, lots of trouble, but also lots of fun.

ORIGINAL PAPERS AND DISCUSSIONS.

THE EFFECT OF TIME AND TEMPERATURE ON THE MICROSTRUCTURE OF PORCELAIN.[1]

By Albert B. Peck, Pittsburgh, Pa.

Introduction.

Many investigators have studied the microstructure of porcelain with regard to the development of the several constituents, particularly the sillimanite at different temperatures, but in all the literature on the subject there appears to be no record of any well-defined effort to correlate the time effect on the sillimanite and quartz with that of temperature. This assumes some importance in view of a recent contention that the constitution and microstructure of porcelain bodies is dependent chiefly upon the temperature of burning and in only a relatively small degree upon the differences in length of burn as will be found in the practices of individual concerns.[2] The use of this principle as a guide in the prediction of the burning temperature of commercial bodies by a microscopic examination has brought forth criticism from Mellor[3] who argues that the evidence as advanced by him tends to show that extended time can do the work which requires a higher temperature with a shorter time and that sillimanite crystals develop much more freely in certain mixtures than in others composed of the same constituents but differently proportioned. The development of sillimanite, therefore, can not be used as an indicator of the burning temperature.

The statement that the time element is relatively small in

[1] By permission of the Director, Bureau of Standards.

[2] Bur. Standards, *Tech. Paper* 80, 33; *Trans. Am. Ceram. Soc.*, 18, 377–423 (1916).

[3] *Trans. Eng. Ceram. Soc.*, 16, 71 (1916–17).

importance as compared to that of temperature strikes one immediately as surprising since it is in direct opposition to well-established facts. The Geophysical Laboratory has shown that impalpable amorphous powder of CaO, obtained by the calcination of $CaCO_3$, when heated for a week at a temperature of 1300–1400° C (1000° below its melting point), results in a product which is crystalline and resembles, in all respects, CaO which forms out of melts of different compositions. This principle is much used by them in their work to obtain in crystalline form compounds which are unstable at their melting point or to increase the size of crystals for microscopic examination. If this is true in such silicate work, it seems reasonable to suppose that increased length of burning should produce marked effects on the microstructure of porcelain.

Moreover, porcelain represents a system which is in incomplete equilibrium. The nearest approach to equilibrium in porcelain is in high-fired bodies consisting of glass and sillimanite. There may be a question whether this represents true equilibrium but there can be no doubt that ordinary porcelain bodies represent incomplete equilibria because the reactions between the constituents can be observed as they take place, stopped or prolonged simply by varying the elements of time and temperature. It appears, therefore, that the longer the action takes place, the nearer should it approach an equilibrium and the greater should be its difference compared with a body not subjected to the action for so long a time. Temperature is of great importance but it seems possible that the time factor as it varies in practice may also play an important part in the resulting constitution and microstructure.

Present Investigation.

With these facts in mind it was deemed worth while to make a few burns upon some quartz-clay-feldspar bodies of known composition under known conditions of time and temperature controlled as accurately as possible, with the purpose of determining by microscopic study how the increase in the time of burning affects the solution of the quartz and the development of sillimanite. This also led to a study of the manner of the formation

of sillimanite in porcelain and the presentation of a new theory as to its development during firing. Unfortunately it is impossible with present means of microscopic examination to place on an exact quantitative basis the amounts of the quartz solution and formation of sillimanite and all descriptions of the constituents of porcelain must necessarily be relative in character.

Composition and Burning.

The body used had the following composition:

	Per cent.
Ball clay	10
Georgia kaolin	35
Feldspar	18
Flint	37
	100

The burns were carried out in the following manner: The bodies were put through a regular draw trial burn with a temperature gradient of 20° C per hour above 1150° C. Samples were taken for purposes of study from those drawn at 1290° and 1350°. These were later burned again, the temperature being raised to 1290° and 1350°, respectively, and held there for 5 hours. Samples were again taken and the burns repeated under conditions as nearly as possible like the preceding. In all, three burns were made exclusive of the draw trial firing. The total length of time the bodies were held at 1290° and 1350° was 15 hours.

Description of the Microstructure.

Specimen Drawn at 1290° C, Fig. 1.—All the clay has dissociated. The outlines of the larger original clay grains still show; some are composed of sillimanite needles and others appear to consist entirely of the amorphous sillimanite. The smaller clay grains appear to have been dissolved since none can be seen. The quartz is sharp cornered and shows no evidence of solution.

Five-hour Burn, 1290° C, Fig. 2.—In this body the outlines of the clay grains as a rule are not so definite and they appear to have been attacked by the feldspar. Dissociation is complete. Sillimanite is somewhat better developed but, due to the small size

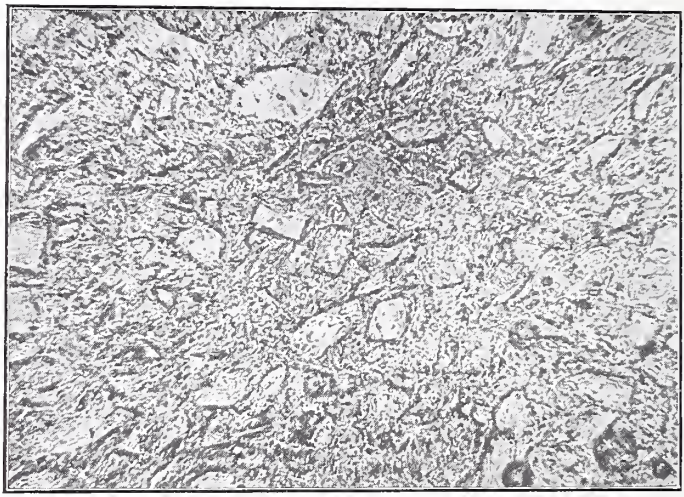


FIG. 1.—Draw trial, 1290° C. Sillimanite is very poorly developed and no solution of quartz has taken place, all grains possessing sharp corners. Magnification 300 diameters.

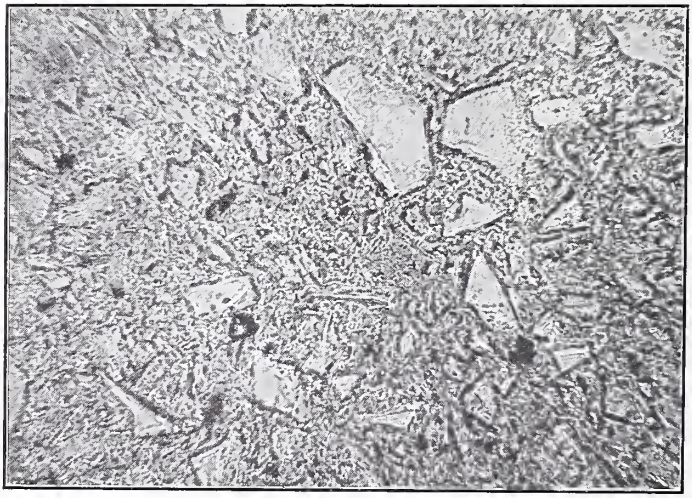


FIG. 2.—Five-hour burn, 1290° C. Sillimanite is still rather poorly developed. The quartz shows a beginning of solution by the rounding of the corners of the smallest grains. Magnification 300 diameters.

of the crystals and the comparatively low magnification used to better bring out the solution of the quartz, this does not show clearly in any of the photographs. The quartz shows slight rounding of the corners of the larger grains but this evidence of solution is more noticeable on the smaller fragments.

Ten-hour Burn, 1290° C, Fig. 3.—The ground-mass is composed of feldspar glass thickly filled with very minute crystals of sillimanite which are difficult to resolve except with high magnifications. Indefinite outlines of what were original clay grains can occasionally be seen but for the most part they have lost their shape entirely and have been dissolved in the feldspar

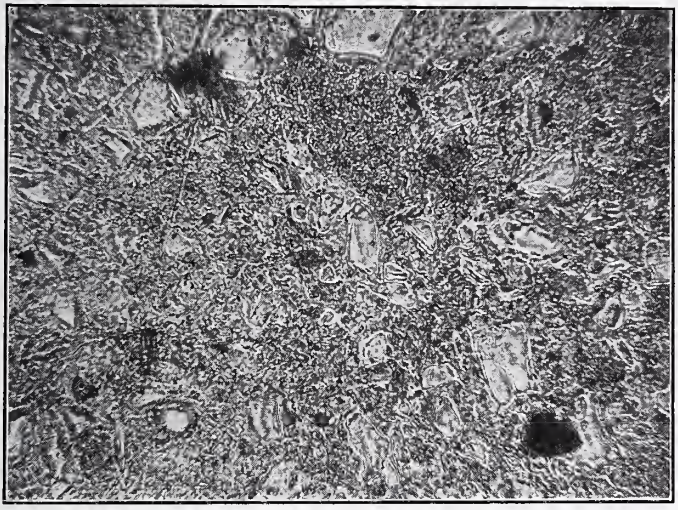


FIG. 3.—Ten-hour burn, 1290° C. The sillimanite crystals have increased in size and amount. Quartz solution is also greater. Magnification 300 diameters.

glass. When observed they are usually composed of sillimanite crystals larger than those of the ground-mass. The quartz is noticeably dissolved and each grain is surrounded by a narrow zone of glass. Even the largest grains are rounded on the corners and edges and it is particularly noticeable on the smaller ones, some of which have almost entirely disappeared.

Fifteen-hour Burn, 1290° C, Fig. 4.—The ground-mass is again composed of sillimanite crystals embedded in glass and they are slightly larger than those in the previous body. Groups of crystals showing the outlines of the original clay grains are very rarely met with. The condition of the quartz, the wider zone of glass and greater rounding of the fragments, gives evidence

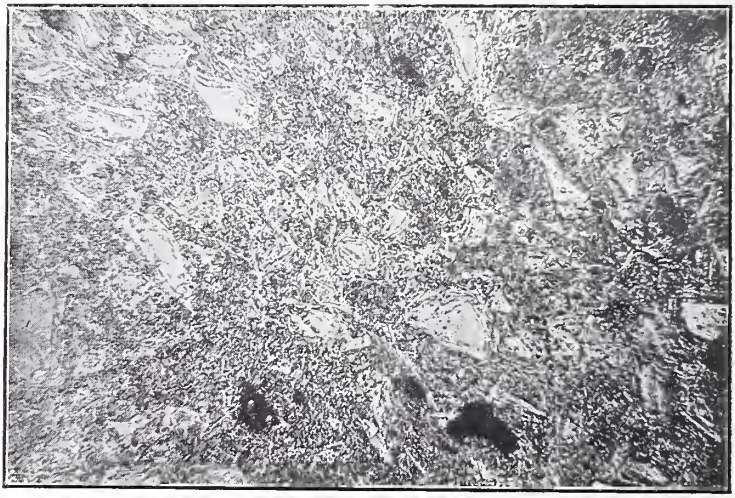


FIG. 4.—Fifteen-hour burn, 1290°C. A great increase in the amount and size of the sillimanite crystals and the amount of quartz solution over that of the ten-hour body. Magnification 300 diameters.

of greater solution than had taken place in the ten-hour firing. A comparison of Figs. 1 and 4 gives clear evidence of the change increased length of burning will cause in porcelain bodies. This is even more strikingly brought out in the series burned at 1350° C.

Specimen Drawn at 1350° C, Fig. 5.—The clay grains in this body have dissociated and have lost their outlines almost entirely. The ground-mass shows glass containing very small crystals of sillimanite almost so minute as to appear amorphous. A few aggregates of fairly large crystals are present. The quartz shows noticeable solution, having slightly rounded corners and a very narrow zone of glass surrounding each grain.

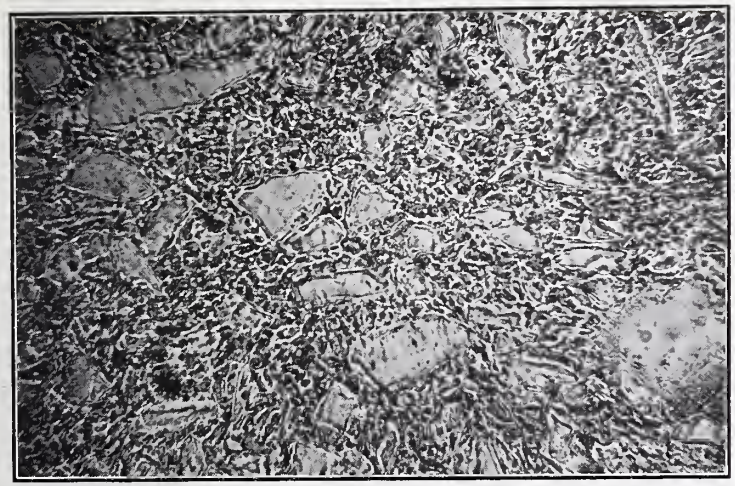


FIG. 5.—Draw trial, 1350° C. Sillimanite is fairly well developed and quartz solution has begun. Magnification 300 diameters.

Five-hour Burn, 1350° C, Fig. 6.—This burn shows an increase in the size of the sillimanite crystals over those of the previous body. The increased solution of the quartz is also apparent

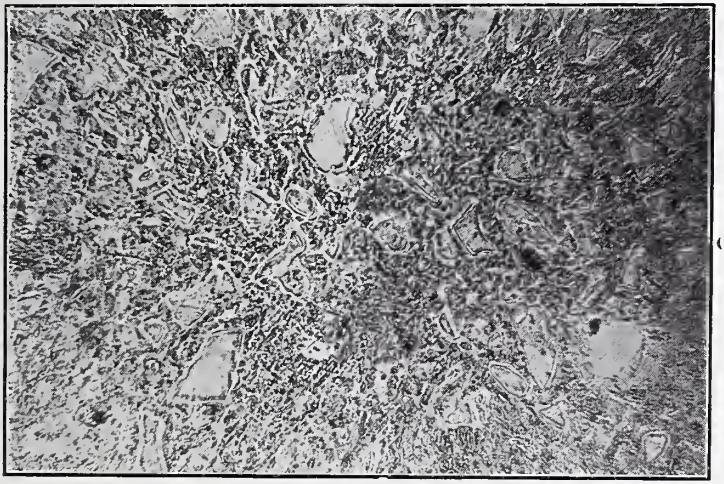


FIG. 6.—Five-hour burn, 1350° C. Note the great similarity of this body to that shown in Fig. 3. Magnification 300 diameters.

from the more pronounced rounding of the corners of the grains, the slightly wider zone of glass around them, and the fact that some of the smallest grains have almost entirely been dissolved. Comparison of this burn with that of Fig. 3 (ten hours at 1290° C) shows a striking similarity particularly in the solution of the quartz. The similarity between development of the sillimanite is also close but not quite as apparent as in the quartz solution. In the latter case ten hours' firing at a lower temperature has accomplished the work of five hours at a higher temperature.

Ten-hour Burn, 1350° C, Fig. 7.—The ground-mass is composed of glass with sillimanite crystals which are larger than those of the five-hour burn at 1350°. The whole section has a more

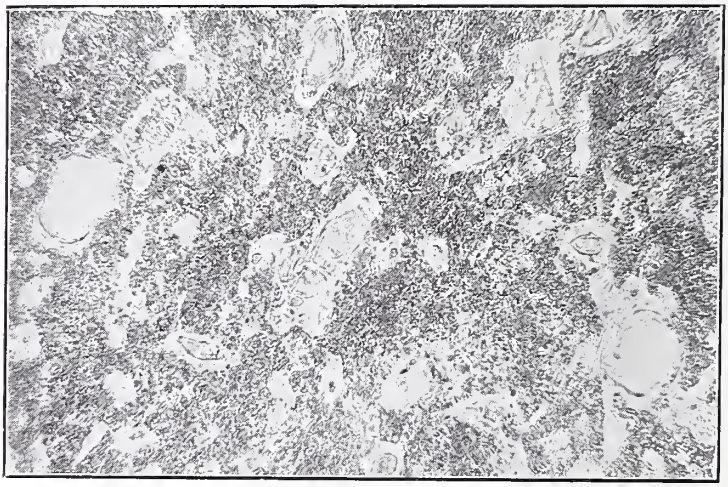


FIG. 7.—Ten-hour burn, 1350° C. The quartz is very much dissolved and much more glass is present. Magnification 300 diameters.

glassy appearance—due to the large amount of quartz which has gone into solution. A few aggregates of larger crystals of sillimanite indicate the position of original clay grains. The most striking feature is the amount of solution of the quartz. Every remaining grain, even the largest, is much corroded and surrounded by a wide zone of glass. In many instances the grain has been entirely dissolved leaving only a patch of glass in its place.

Fifteen-hour Burn, 1350° C, Fig. 8.—Again the sillimanite crystals show an increase in size and no trace of original clay grains remains. The quartz has almost entirely dissolved; only the remnants of the largest grains can be seen. How great this solution has been is well shown by a comparison of Figs. 9 and 10. These fields are identical with those of Figs. 5 and 8, with the exception that crossed nicols have been used to bring out more

FIG. 8.—Fifteen-hour burn, 1350° C. Note the still greater increase in quartz solution. Sillimanite crystals have also increased in size. Magnification 300 diameters.

clearly the quartz. When it is considered that the body represented in Fig. 9 contained 37 per cent quartz and that this has been reduced to about 5 per cent in the body shown in Fig. 10, the difference having dissolved in the feldspar glass, the variation in microstructure which results from differences in length of burning is strikingly brought out.

Comparing the microstructure of these two series of burns, one would hardly suspect that any two of them were burned at the same temperature, except those represented in Figs. 3 and 6, and in this case it is a longer fired, lower temperature body which almost duplicates a shorter fired, higher temperature body. As a consequence it seems doubtful whether the temperature

of burning could be estimated from the microscopic examination of a porcelain.

In passing, attention is called to the fact that slight amounts of fluxes such as CaO and MgO, as shown by Riddle,[1] cause marked variations in microstructure. This reflects itself in the

FIG. 9.—The same field as shown in Fig. 5 but taken with crossed nicols. Magnification 300 diameters.

increased translucency of the body, increasing flux producing greater translucency.

Possible Effect on Translucency.—Translucency of porcelain appears to have a direct relation to the proportion of glass and sillimanite in the body. Microscopically, all highly translucent bodies show glass and sillimanite as the chief components with perhaps a little quartz. It has been shown that increased length of burning results in an increase in the amount of glass and sillimanite and a decrease in the quartz. It seems possible, therefore, that the translucency of ware might be increased by firing for longer periods, particularly at temperatures upwards of 1300°.

The amount of feldspar used also probably affects the translucency, not only by providing more feldspar glass itself, but also

[1] Private communication.

FIG. 10.—The same field as shown in Fig. 8 but taken with crossed nicols. This serves to show the great amount of quartz which has dissolved during the fifteen hours of burning although the temperature remained constant. Magnification 300 diameters.

more of a medium for faster solution of the clay and crystallization of sillimanite and solution of the quartz.

The Effect of Ordinary Differences in Time of Burning upon the Microstructure.

The preceding pages have shown that time markedly affects the constitution and microstructure of porcelain. The question remains, whether ordinary differences in burning practice are sufficient to cause differences in microstructure.

Ordinarily this depends on two factors—the final temperature to be attained and the size of the kiln. Obviously, the higher the final temperature, the longer will it take to reach that temperature, using the same gradient in each case.

According to European practice, in small kilns the burns may range from 18 to 28 hours, in large kilns from 30 to 45 or 54 hours. In Fig. 11 are shown two heating and cooling curves of commercial kilns both fired to the same temperature—about 1320–1330°, the smaller requiring 52 hours and the larger 59 hours.

It is in the vicinity of 1250° that the microstructure of porcelain bodies of ordinary composition begins to show phenomena other than the melting of the feldspar and the dissociation of the clay into amorphous forms of silica and sillimanite. At this temperature crystalline sillimanite appears in small amounts and at slightly higher temperatures solution of the quartz starts.

Considering the points on the diagram where the heating and cooling curves cross the 1250° ordinate, it is apparent that approximately 21 hours have elapsed in the case of the longer burn from the time 1250° was passed on the upward curve to the time

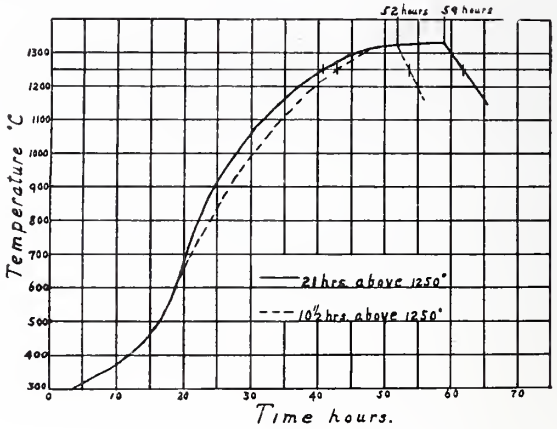


FIG. 11.

it was again reached on the downward curve. Similarly in the second case the elapsed time was about $10^1/_2$ hours, a difference between the two of about $10^1/_2$ hours. During this time the body was in what may be termed the zone of reaction and was, therefore, undergoing changes in structure and constitution. This action while not constant in velocity but varying with the temperature is nevertheless continuous whether the temperature is rising or falling. On account of the flattening of the tops of the curves, the average temperature of the body while in this zone of reaction is probably not far from 1300°. In view of what was found in bodies held at constant temperatures it would not be surprising to find that porcelains, burned according to such

curves as those shown in Fig. 11, exhibited noticeable differences in structure.

Figures 12 and 13 show the microstructure of two porcelains burned in actual commercial practice according to these curves. Each is slightly shorter, that of Fig. 12 requiring 49 hours while that of Fig. 13 required 56 hours, a probable difference in elapsed time of about 8 hours.

The porcelain shown in Fig. 12 shows almost no solution of the quartz. The sillimanite is nearly all present in a crystalline

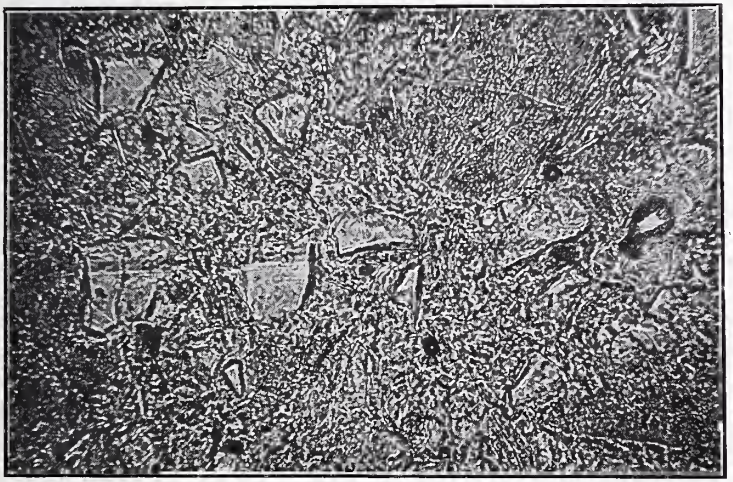


Fig. 12.—The quartz grains are sharp and angular for the most part and the sillimanite is usually found in very minutely crystalline aggregates as shown in the upper right hand corner. Magnification 300 diameters.

state but occurs usually in groups of very minute crystals. The fluxing action of the feldspar has not been marked since the outlines of the original clay grains are frequently well preserved and only small amounts of well crystallized sillimanite are present. The porcelain of the longer burn (Fig. 13) shows greater solution of the quartz as is evident from the rounding of a greater number of the quartz grains. The frequency of aggregates of very finely crystalline sillimanite has become less and more groups of well developed crystals embedded in the feldspar glass can be ob-

served. The contrast between the two bodies seems to be sufficient evidence that the difference in length of burning in practice actually does produce important differences in structure and that the estimation of the temperature should be considered rather doubtful.

The Formation of Sillimanite in Porcelain.

In the study of porcelain the question naturally arises—how is the sillimanite formed? Since no sillimanite enters into the

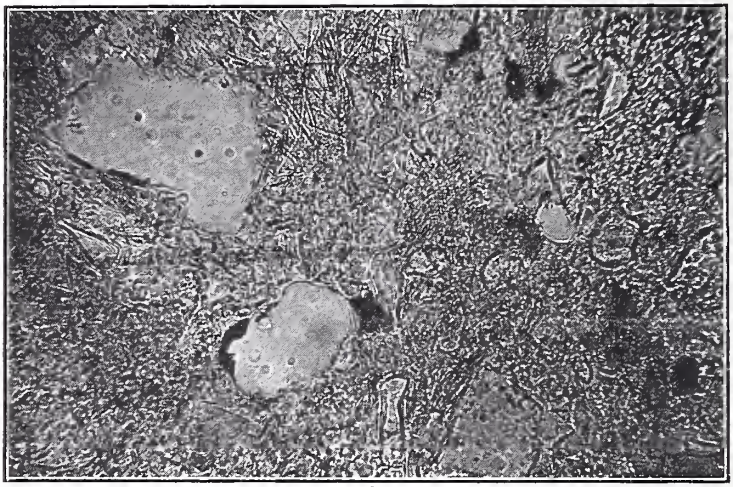


FIG. 13.—In this longer-burn specimen the quartz grains are more generally rounded showing the effects of solution while the sillimanite is more frequently present as large, well-developed crystals in the feldspar glass. A group of these appears near the top of the photograph. Magnification 300 diameters.

composition of the original mix, it must form either through the breaking down of one or more of the constituents of the mix or by a re-combination in some manner of the elements composing it.

Microscopic examination of porcelain reveals the fact that there are really two types of sillimanite present. The first type is shown in Fig. 14[1] and is frequently characterized by a fibrous

[1] Figs. 14 and 15 have been taken from Bur. Standards, *Tech. Paper* 80, by A. A. Klein.

appearance brought about by long, slender crystals orienting themselves parallel to each other, or it may take the form of confused aggregates of almost sub-microscopic amorphous-like grains, or of small, interlocking, poorly formed crystals. Sometimes the outlines of the original clay fragments are retained although the feldspar has usually attacked them and rounded the corners. There seems to be little doubt that this type has been developed by the direct dissociation and molecular change of the clay under

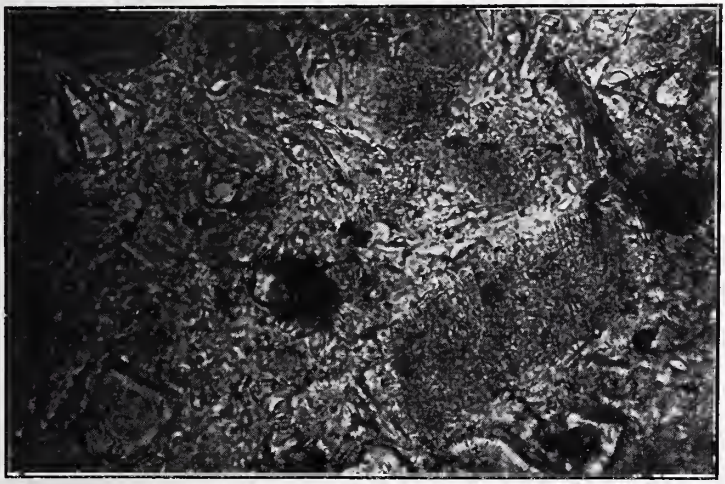


FIG. 14.—In the upper central part are two aggregates of apparently amorphous sillimanite, toward the right hand margin is a fibrous aggregate, and in the lower right hand corner is a group of very small, interlocking sillimanite crystals. Magnification 380 diameters.

the influence of heat alone into amorphous forms of silica and sillimanite. If the action of the heat ceases, the sillimanite remains amorphous, but if continued or increased, the amorphous grains undoubtedly coalesce and gradually form crystalline aggregates in the same manner as does amorphous CaO under the influence of heat at a temperature of 1300–1400° C, 1000° below its melting point.

The second type is shown in Fig. 15 and is represented by definite, well-formed crystals embedded in the feldspar glass.

Their position and development give strong indications that they have crystallized out of solution. These crystals are most abundant in high-fired or high feldspar bodies or may be found projecting from a body into the glaze where molecular mobility is easy and great. They are also observed in long-fired, medium temperature bodies. These crystals represent what may be termed the end product of sillimanite crystallization since they appear to be the only product besides glass in hard porcelain.

Investigators have held different views regarding the formation of this type of sillimanite—some that it is due to devitrifica-

FIG. 15.—In the upper right and lower left hand corners are seen many large, well-developed sillimanite crystals embedded in feldspar glass. Magnification 380 diameters.

tion and others to molecular change of the clay. In order to arrive at some conclusion concerning this, fusions of feldspar and clay in various proportions were made in unglazed porcelain crucibles in a small surface combustion furnace. The fusions were held at a temperature of 1400° for three hours and then quenched by dropping into water. The compositions of the fusions were as follows:

Feldspar.	Clay.
90	10
80	20
70	30
60	40
50	50

The clay in each case was composed of one part of Kentucky ball clay and four parts of North Carolina kaolin. Maine feldspar was used and one fusion of this alone was made at the same time.

Examination of the fusions after quenching showed that the feldspar containing no clay had melted to a clear glass with a refractive index of 1.485 ±0.003. The small amount of original quartz had practically all disappeared as was the case in all the other bodies. No other components could be found. The body containing 10 per cent clay was composed almost entirely of a glass with an index of 1.490 ±0.003, slightly higher than that of the pure feldspar glass. Besides residual quartz there was a very small amount of minute sillimanite crystals. These usually occurred in small aggregates with individual crystals distinctly separated from each other. Their outline was definite in all cases although the end terminations tended to be rounded rather than sharp. With 20 per cent clay the glass increased its index slightly to 1.493 ±0.003. The amount of sillimanite crystals was slightly greater than in the previous body and their general appearance was the same although occasionally a few larger and more definitely terminated crystals were present. At 30 per cent clay, the refractive index of the glass was identical with that of the previous body. The amount of sillimanite crystals had increased but their general appearance was the same. The same held true for the bodies with 40 and 50 per cent clay; the refractive index of the glass remained the same and the sillimanite crystals showed a steady increase in amount. No undissolved clay was present in any of the foregoing bodies.

Following this, two mixtures of feldspar containing 20 per cent and 30 per cent clay were fired to cone 14 in a large downdraft kiln along with some ware and allowed to cool down with the kiln. Subsequent examination of these bodies showed that

they differed in no respect from the bodies which had been quenched. The refractive index of the glass was 1.493 ± 0.003, the crystals of sillimanite were the same size, and, as far as could be judged, were present in the same amounts as in the respective quenched bodies. Neither of these two fusions showed large well developed needles of sillimanite as would be expected if the sillimanite were formed by devitrification of the feldspar glass on slow cooling. Since we find the development of the crystals the same in both quenched and slowly cooled bodies, the obvious conclusion must be that the sillimanite forms as such at the temperature of the porcelain kiln and not through devitrification on cooling. If the latter were true, we would expect to find an increase in the amount of sillimanite crystals and probably an increase in their size and perfection of development under slow cooling. Furthermore, we would expect the feldspar glass to show a somewhat lower index of refraction—due to its loss of Al_2O_3 through increased sillimanite crystallization.

In view of the facts as shown in these experiments the conclusion must be that the sillimanite in the feldspar glass and probably also the well developed sillimanite crystals in porcelain are formed at porcelain kiln temperatures and are not due to devitrification.

Exactly how the crystallization of the sillimanite takes place is not easily determinable. Zoellner[1] has shown that feldspar will dissolve 3.5 per cent Al_2O_3, 14 per cent clay, and 60–70 per cent quartz, the relations not being changed in the presence of more than two components. Referring to the feldspar-clay series described, it is apparent that the index of refraction of the feldspar has reached its maximum at a point between 10 and 20 per cent of clay content and that beyond 20 per cent the only change is an increase in the amount of sillimanite formed. Once the feldspar glass has become saturated with regard to Al_2O_3, conditions become very favorable for crystallization of sillimanite, which might take place in several ways. Kiln temperatures vary over a range of several degrees and a slight drop in temperature would very probably result in the beginning of crystallization. It has been previously pointed out that clay

[1] *Sprechsaal*, 41, 471–73, 533–36.

passes into an amorphous phase of sillimanite and later into a crystalline phase. When the crystalline sillimanite has once been formed in this manner in a saturated solution it may immediately serve as a nucleus for further crystallization. After crystallization has started there would be four constituents present—saturated feldspar glass, amorphous sillimanite, silica resulting from the breakdown of the clay, and crystallized sillimanite. It is commonly known that if into a saturated solution, out of which small crystals are crystallizing, a large crystal of the same kind is introduced, the large crystal will under the same conditions continue to grow at the expense of the smaller ones until finally they may have disappeared completely and have been recrystallized as part of the large crystal. If several large crystals are introduced instead of one the result is again the same number of still larger crystals. Amorphous bodies are also more soluble than crystalline bodies of the same chemical composition. Therefore, where there are present saturated feldspar glass, amorphous sillimanite and a little crystallized sillimanite, it appears quite probable that the amorphous sillimanite passes into solution in the feldspar glass just as fast as aluminium silicate passes into the crystalline state and crystallizes as sillimanite, around nuclear crystals which were formed either by fluctuations in temperature or by coalescence of amorphous into crystalline sillimanite. While conditions remain reasonably constant the reaction would proceed in the foregoing manner until, if firing does not cease, all amorphous sillimanite would be transformed into saturated feldspar glass and crystallized sillimanite.

This is precisely the condition found in feldspar-clay mixtures or in long- or high-fired porcelain except that in the latter two cases some undissolved quartz may remain. It seems quite probable, therefore, that sillimanite forms as well-defined crystals in porcelain at kiln temperatures and not by devitrification.

The size of the crystals developed appears to be influenced by three factors—the amount of feldspar or other fluxes present, the mobility of the feldspar as affected by the temperature of firing, and the length of time of firing. It is hoped that further work will throw more light on these factors.

Summary.

Petrographic microscopic examination shows that quite dissimilar bodies can be produced by holding porcelain at a constant temperature for lengths of time which lie within the variations of commercial practice, the clay passing into amorphous silica and sillimanite which in turn passes into crystallized sillimanite. The solution of the quartz takes place at the same time. The difference in structure becomes more marked, the higher the burning temperature.

The longer the burning at a constant temperature, the greater is the amount of sillimanite formed, the larger and better developed do the crystals become, and the greater is the amount of quartz dissolved by the feldspar.

Bodies fired for longer periods at lower temperatures have been produced, which simulate very closely those fired at higher temperatures for shorter periods.

Long burning tends to produce increasing amounts of glass and sillimanite and, therefore, probably increases the translucency.

Sillimanite crystals form; first, by a gradual passage from the amorphous to crystalline state without the aid of a flux; second, with the aid of feldspar as a flux, they form in well developed crystals in the feldspar glass at kiln temperatures through solution and recrystallization of amorphous sillimanite.

In view of all the preceding facts it does not appear that the temperature of burning of porcelain bodies can be predicted from a petrographic microscopic study without at least some knowledge of composition and length of firing.

The author here takes pleasure in expressing his sincere appreciation to Mr. A. V. Bleininger, Mr. F. H. Riddle, and Mr. J. W. Wright for aid and advice during the progress of the work.

THE DESIGNING OF FACTORY LAYOUTS FOR THE CLAY INDUSTRIES.

By T. W. Garve, Columbus, Ohio.

Technical designing is an art as well as a science. Any design or construction which has to perform a certain duty during its time of usefulness is a creation, but only worthy of that name if the most effective solution has been found, and the most effective is usually the simplest. Effectiveness, simplicity and ornateness are fundamentals for the factory designer and their importance is in the order named.

The designing of public buildings and dwellings has always been the work of the architect, but when it came to the design and construction of skyscrapers, with the abundant use of steel and reinforced concrete and power equipment, the work had to be subdivided. The architect first works out the plan, then the engineer designs the frame, and finally the artist embellishes the structure. In designing factories the engineer's work is of chief importance.

Housing is the sole purpose of the building, and the engineer's work, though important, is secondary. He first places the machinery in every detail and then adds the structure to support and house it. The architect and artist are not essential, but in many instances it is to be regretted that the latter is not consulted.

The architect's work is decidedly varied but his problem is a single one, namely, housing. The engineer's work is very different; he is dealing with manufacturing processes and must be an expert in each process for which he designs a factory, or he must call to his aid those who are experts to work out the details of each problem, leaving for him the general mechanical features and final assembly of the factory as a whole.

A machine is usually built first as a trial, then corrected and improved upon, and often completely remodeled and rebuilt before it is turned out in a large number as a type. A factory,

however, cannot be corrected so easily. Buildings cannot be shifted nor can foundations be torn out; there is, besides, too much money invested in a manufacturing plant to permit of experimenting afterwards; it has to be *right in the first place.* How many clayworking plants have been right in the *first* place?

Of course, a clayworking plant is not a shoe factory or an automobile factory; there are no two clayworking plants alike—hence the very essential requirement of the designing engineer is to be familiar with the branches of clayworking and to be broad enough to study the proposition in question from every angle.

We cannot do better than quote from Day's "Industrial Plants,"[1] as follows:

> "It will be evident that during the limited period needed for the designing of a new plant, almost every branch of engineering may be called into play. Those for whom the new plant is being built must be counted upon to dictate primary requirements, but in the majority of cases their experience should be supplemented by the broader knowledge of others.
>
> For the need of a better term 'industrial engineering' has been quite generally adopted as the name for the professional service rendered by independent engineers engaged upon such work.
>
> The industrial engineer coördinates the mass of technical notes turned in to him by the various engineers engaged upon his work, and on account of his intimate acquaintance with his client's needs, aims, and resources, he is able to consider the conclusions presented from a broad standpoint that is wholly unknown to the individual workers. He is to remember constantly that it is the final result that measures the usefulness of his efforts, and that it is absolutely immaterial from what source a given suggestion may come. As a general proposition, complete plans and specifications, covering all features entering into building structures, should be prepared prior to soliciting bids; but this work must, of course, be done by men thoroughly conversant with all available standards and the usual practices adopted in shops."

Any clayworking proposition should be investigated very thoroughly before a preliminary plant layout is studied and worked out. The kind and quantity of ware to be made, the physical conditions of the clay, and local conditions and requirements will determine the kind of machinery to be used and the type of dryer and type of kilns to be adopted. There is a great variety of machinery of different types and makes on the market

[1] "Industrial Plants," by Charles Day, The Engineering Magazine, New York, N. Y.

and it is often difficult to select the equipment best suited. The location of the plant, market conditions, quantity of clay and the capital available will be factors in determining the kind of building materials to be used (whether brick, steel, lumber, concrete, kind of roofing, etc.).

We have heard much in these days about conservation of fuel and to this end kiln and boiler furnaces and methods of firing have been improved. How much fuel, however, is daily being wasted in a factory where the routing is not the shortest possible, or where conveyors are too long, elevators too high, and where power transmitting appliances of relatively low efficiency are abundantly in evidence. We quote Lovejoy from "Economies in Brickyard Construction and Operation" as follows:

> "From the power house we go into the factory and find short drives with belts drawn up by stretchers, unnecessary countershafting, and both line and countershafting out of line, crooked and badly wobbling, top pulls on belts and unnecessarily crossed belts—the list is too long to enumerate, but everything dragging and eating up boiler fuel."

In this paper we have attempted to point out some essential, general features in clay working factories without, however, taking up variations in each type, the discussion of which would be endless.

Clay Haulage.—The distance the clay is to be hauled should be as short as possible. Whether the plant is to be built close to town or close to mine or clay bank is often a local consideration and cannot be stated definitely one way or the other; we would have to know the situation of the market, the railroad, and other local peculiarities such as fuel, labor, water, housing, transportation, etc.

If a mine shaft is used close to the plant, the clay or shale should be delivered from it at the highest point of the factory in order to eliminate further elevating as far as possible. Gravity haul from the bank is recommendable if the site of the plant and the elevation of the bank will permit. Most often the clay is to be elevated and a winding drum or steam hoist is used at the highest point of the incline. Since clay dump-cars are made of the bottom, side, and end dump types, there is much latitude

in conveniently arranging the clay storage and clay feeder or any other preparing machine to be fed. Any incline should be steep enough to allow the empty cars to return with dragging cable. If the clay or shale is to be hauled in by wagons or railroad cars, it may be advisable to build up a slight incline, by earth filling or by trestle, to avoid a great depth below ground for clay hoppers, feeders, or elevator tail ends.

Clay Storage.—Raw clay is stored for the purpose of sufficient supply during winter or during bad weather; to help over any breakdown at the mine or bank; to secure a better and more uniform mixture; and to improve the working condition of the clay and quality of the ware.

Clay storage equipment should be an independent feature of the plant that can be in use continuously or intermittently as conditions require so that all unnecessary rehandling is avoided. It should face the clay bank and later extension should be possible.

Ground clay storage within the factory is an essential feature to insure continuity so that the consequences of a breakdown, or any irregularity in supply of clay, will be checked at the bin and not be felt throughout the succeeding manufacturing processes of the plant.

Machinery Building.—One end of this building, where the clay enters, should be in touch with the clay storage building and the other end close to the dryer (or kilns). The shortest possible routing and as automatic as is practical is to be devised avoiding, however, a too elaborate system of conveying machinery. If it can be worked out it is best to do all the elevating in the first place and then to let the clay pass from machine to machine by gravity. If two or more sets of preparing machines are used, provision should be made for a certain elasticity in the arrangement by which an exchange of duty can be accomplished. This applies especially to large plants which turn out two or more kinds of ware and where a corresponding number of types of preparing machinery is to be installed. Here the grinding or preparing outfit for one kind of ware may be called upon to do duty for the other kind of ware and vice versa. Other demands may arise later during the manufacture and development of the

product and there are often quite a number of eventualities which have to be taken into consideration in designing the machinery layout and building.

If electric motors are employed in power transmission they should be driven by belt to machinery and by belt or silent chain to shafting, avoiding gears. The shafting should be well balanced and placed so that the drives are of proper length and the lower side of every belt is pulling—avoiding bevel gears and cross belts as much as possible. A quarter twist belt, if installed properly, is not objectionable for light duty, especially if it enables a more satisfactory arrangement of machinery.

There should be ample space around each machine, without waste, although too much is better than not enough. There should be sufficient head room above each machine for a chain block and suitable structure to carry it.

The light question is an important consideration often overlooked or neglected in clay working plants.

Avoid deep pits if possible and always keep above ground water level with the bottom of any pit.

Line shafting should be carried on independent concrete or brick piers and all machinery platforms should be sufficiently heavy and well braced to reduce building vibrations to a minimum.

The conveying machinery usually employed in clay plants consists of troughing belt conveyors and belt bucket elevators. Sticky or wet clays are very difficult to convey and elevate, even on inclined elevators with open buckets or flights, and should be avoided if possible.

If depth or elevation can be had, it is advisable to use a gravity hopper below the dry pan instead of scrapers—thus lessening maintenance cost and power consumption.

The dry grinding plant should always be separated from the rest of the factory by a dust-proof wall or partition. Dust is harmful to human lungs as well as to bearings and will cause belts to slip. Shafts can pass through such partitions but belts should not go through. Modern factories sometimes encase the grinding machines and remove the dust by fan suction.

Finally, provision should be made for expansion in every factor of the plant for future increase in capacity.

Power House.—The power equipment should be independently enclosed in a dust and fireproof building or rooms. If belted to a line shaft it should be so located as to balance the load, or in any event, there should be no material extension of the line shaft to connect with the power drive and an end connection should be

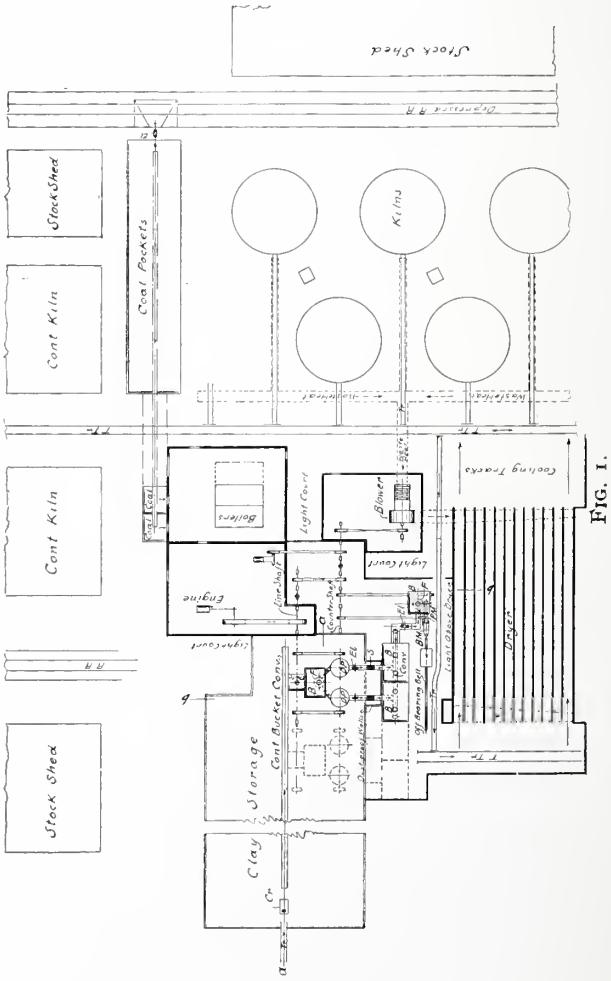


FIG. 1.

that nearest the heaviest load. Where steam is used for drying, the equipment for such applications should be close to the power plant in order to keep down the cost of installation, to conserve the heat, and where it will be under the watchful eye of the power operator, bearing in mind, of course, that the position of the dryer relative to the movement of the ware is of first importance.

There should always be a surplus boiler, enough room left

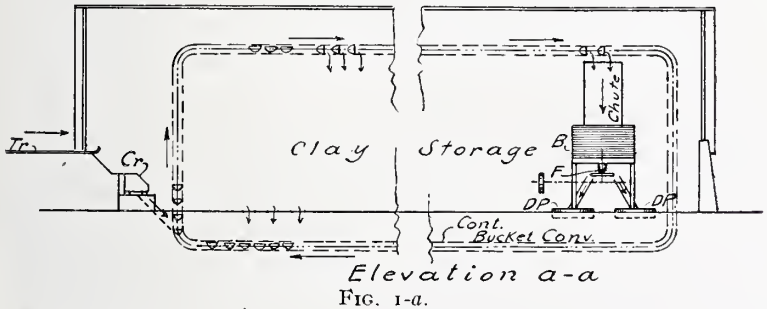


FIG. 1-*a*.

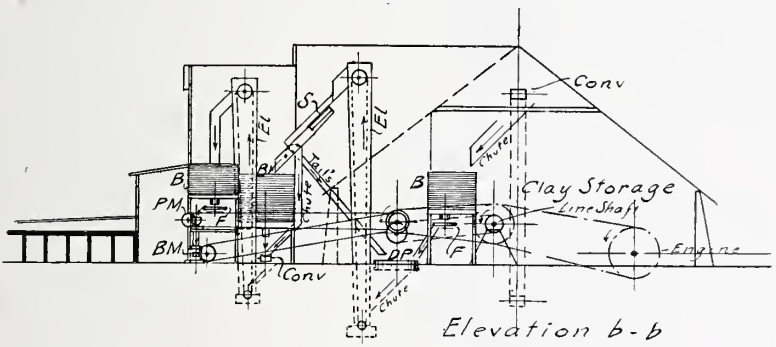


FIG. 1-*b*.

at the rear for removal and cleaning of boiler tubes, and space at the front to allow a sufficient coal supply close to the furnaces. The boiler house should be so located that fuel can be delivered to the boilers in a convenient and economical manner.

There should be sufficient space between engine cylinder and building wall to allow the removal of piston and rod.

The power house should be fireproof and provision made for future extension.

Dryer.—The receiving end should be close to the machinery delivery and the cooling end should face the kilns. At the end of the dryer toward the kilns cooling space or cooling tracks should be provided. The closer the dryer is to the kilns the shorter

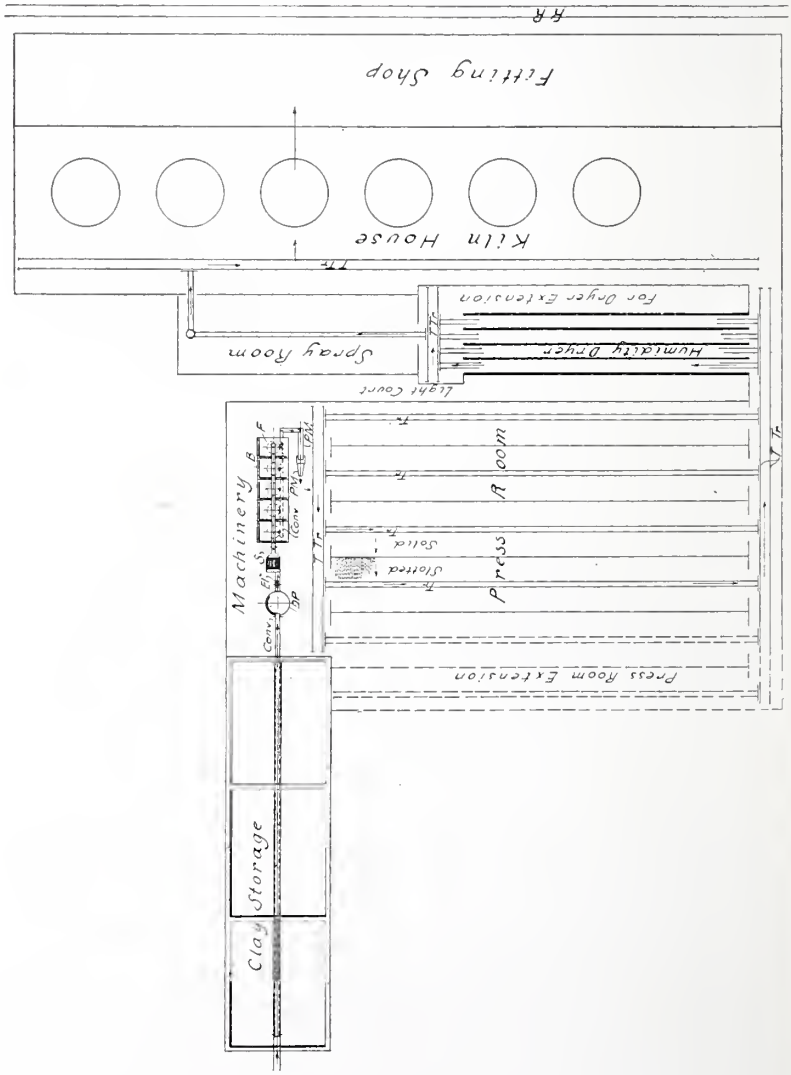


FIG. 2.

the haulage of the dried ware and the shorter the waste heat duct from the kilns, if such a system is employed.

It is imperative that the dryer be fireproof throughout.

Kilns.—Convenient car switching for the ware and convenient delivery and distribution of coal must be possible. The kilns should, without waste of space, stretch along a stock shed and railroad switch.

As an illustration we have sketched the three clay working plants shown in Figs. 1, 2 and 3. For the sake of clearness it was deemed advisable to abbreviate some of the lettering as follows:

B.....	Bin	B. M.....	Brick machine
Cr.....	Crusher	D. P.....	Dry pan
Conv.....	Conveyor	S.....	Screen
El.....	Elevator	Tr.....	Track
F.....	Feeder	T. Tr.....	Transfer track
P. M.....	Pug mill	R. R.....	Railroad

Building Brick Plant (Fig. 1).—The clay storage building can be extended to any desired capacity towards the clay intake end. The crushed clay or shale from the crusher is delivered by a short chute to a continuous bucket conveyor which can supply either the storage building or the pan bin directly; it can also take the stored shale by means of the underground strand up to the pan bin. The screened clay from the pan can chute to storage bins at the bottom of which a belt conveyor is operating towards an elevator at the pug mill bin. If these storage bins are filled, the screened clay can chute down directly to the belt conveyor mentioned. Additional pans and additional storage bins for ground shale can be built without any other change in the arrangement (shown dotted).

The engine is running over, the drive is located in the middle of the line shaft between equal loads and all belts are pulling at the under side.

The dryer blower is driven from the main engine during the day and by a separate motor or gas-engine during the night. The necessary clutch coupling in the counter shaft is provided for.

The loading track along the off bearing belt is short, straight, and close to the intake end of the dryer.

The waste heat intake to the blower is located very near the center of the periodic kilns.

Dryer, blower house, and power station are built with independent brick walls and good light is obtainable. The machinery building will receive its light from two light courts, through windows above the dryer and elevator monitor.

The transfer track from the dryer stretches along one side of the

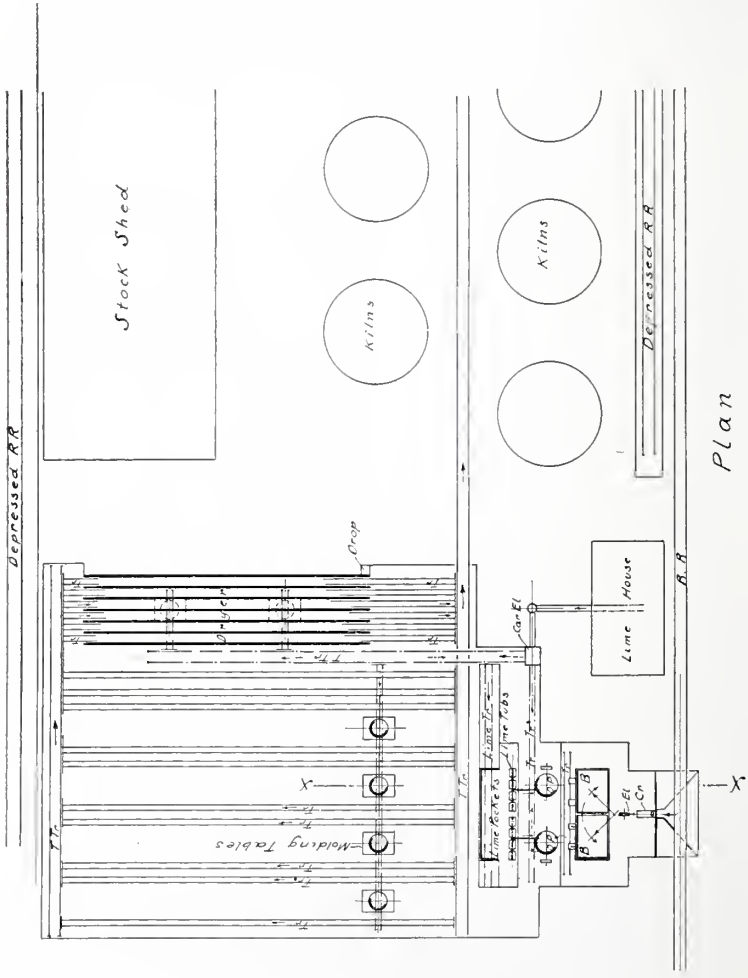


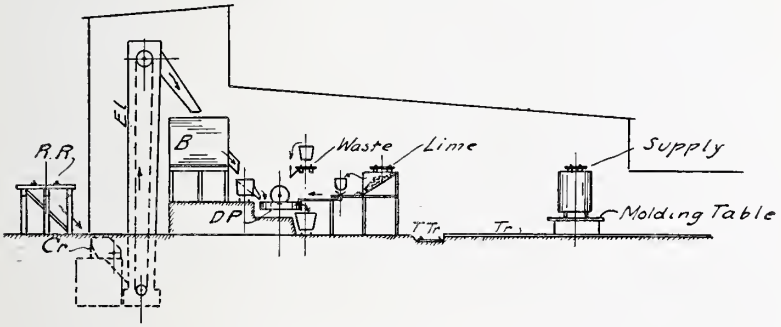
FIG. 3.

periodic kilns and passes between the two rows of tunnels or chambers of the continuous kiln. Electric transfer cars can be used for operating along these kilns. The intake tracks to the kilns are straight and short.

There are two railroad switches and three stock sheds. Coal is being delivered close to the kilns and boilers.

The dryer can be extended and also the kilns.

It is often difficult to find a satisfactory solution for a machinery layout with the application of a steam engine with dependent



Elevation X-X

FIG. 3-*a*.

shafting and belting, especially for a plant of large capacity. With the application of electric power we have much more latitude in the machinery arrangement and less difficulty in finding a satisfactory solution.

Terra Cotta Plant (Fig. 2).—The clays are dumped by car or conveying machinery into storage bins and from there taken by a conveyor and elevator to a dry pan. The ground clay is screened and delivered into small bins for each clay with a feeder at the bottom of each bin and a belt conveyor alongside the feeders to carry the clay to an open-top pug mill, from which it passes down to a closed top or conical pug mill.

The clay is now ready for the molders and is taken to the so-called press building by trucks on wheelways or by cars on tracks

as shown. The clay taken down the first track in the press building is here made into ware which is then moved over towards the second track to remain for a while on a slotted floor, with a few steam pipes below for partial drying, before the ware is picked up from the second track and taken into the humidity tunnel dryer, or the ware can be run directly to the dryer. Over a part of the press room is a second story for storage of molds and for the drafting room.

From the dryer the ware passes through the so-called spray room for the particular coating. The grinding and preparation room (pebble mills, tanks, etc.) for the glazes is located in the second story of this building; this would also be a good place for a laboratory and an office.

Close to the spray room is the kiln house and next to the kiln house the fitting shop and then the railroad. Terra cotta work is, for the most part, special work to suit the requirements of the orders and made up accordingly, hence we have no stock shed or stock room but a fitting shop instead, before shipment is made.

A separate power house for generating electric power is not shown in the drawing.

Silica Brick Plant (Fig. 3).—From the railroad car, on a low trestle, the quartzite is dumped into a pocket in front of a jaw crusher, then crushed, elevated and stored. It is drawn from the bins and run to the pans. From the other side the pans are supplied with the necessary lime. The lime slip is piped down from a row of lime tubs especially made for this purpose (Fig. 3-*a*). Each tub has its independent bottom discharge above the same trough. To one side of these tubs are open lime pockets supplied with lime from cars on the track above. The lime is brought in here by cars on tracks and through an elevator from the lime house near the railroad.

The prepared clay-lime mixture is dumped into cars below the pans and taken to the elevator and then to the molding room by discharging them over cylindrical containers open at the bottom in the center of each molding table. The molding tables are at one end of the room to allow for long tracks at the other end in order to have the ware lose some of its water before it enters the

tunnel dryer. There are also molding tables above the dryer for larger wares which require slow drying.

The car and track shown in elevation above the pans are for the return of all waste to the pans. The lower loading and transfer tracks and the previous elevator are used for this purpose also. Note the same elevation of the three upper tracks (waste, lime, and supply).

BEHAVIOR UNDER BRASS FOUNDRY PRACTICE OF CRUCIBLES CONTAINING CEYLON, CANADIAN AND ALABAMA GRAPHITES.[1]

By R. T. Stull.

The life of a graphite crucible depends upon so many factors that it is difficult, if not impossible, to obtain accurate data from which consistent conclusions can be drawn. The best that can be done is to test a large number of crucibles under a set of conditions as nearly constant as possible and to take a fair average of the results.

The composition of the crucible, the conditions surrounding its manufacture, its size and shape, and its use and misuse in the foundry, all contribute to determine the length of life of the crucible.

It is not difficult to get coöperation with foundries in testing crucibles, but it is difficult and even impossible to educate the workmen up to the point where they can understand the importance of exercising reasonable care and accuracy and to take more than a passive interest in the work. This is especially true in foundries where the workmen are paid on a tonnage output basis, because it matters not to them whether one or ten crucibles are required to produce a definite quantity of metal.

It requires approximately a month's time to make a crucible of No. 60 size or larger and after the crucible arrives at the foundry it requires from three to four weeks to dry it thoroughly at a temperature of from 100° to 110° C.

Thoroughly dried crucibles give greater service than those which are imperfectly dried, as has been pointed out by Gillett[2] and Bleininger.[3] After the crucibles have been taken from the kiln they

[1] By permission of the Director, U. S. Bureau of Mines.

[2] H. W. Gillett, "Brass Foundry Practice," U. S. Bureau of Mines, *Bull.* 73, p. 161, June, 1916.

[3] A. V. Bleininger, "Notes on the Crucible Situation," *The Clay Worker*, p. 531, Dec., 1917.

may take up as much as 5 per cent hygroscopic moisture and its expulsion in the drier is slow. In order that the tests may be comparable, the crucibles should remain in the drier about one month before being placed in the melting furnace. Some of the crucibles tested in the following work remained in the drier for only $3^1/_2$ days, while others received 29 and 30 days' drying.

Inasmuch as it requires a month to make a crucible which should receive a month's drying treatment before it is tested, and may require two weeks' time in transportation between factory and foundry, the process of testing crucibles is slow and as the size of crucibles tested retails at from $7.00 to $10.00 apiece, the process is also rather expensive when a large number of crucibles is tested.

The number of crucibles tested is altogether insufficient to permit drawing definite conclusions and the conditions under which different groups were tested varied so, that only conclusions of a more or less speculative nature can be drawn for the different groups and from different members in their respective groups. The work has just begun, and so far as it has progressed, may be considered as preliminary.

Crucibles Tested.

Four series of crucibles[1] of three different sizes, No. 60, No. 70 and No. 80D, have been made. The crucibles were tested in two different brass foundries designated herein as foundries No. 1 and No. 2.

Foundry No. 1 operates thirty-six natural-draft pit furnaces using a No. 70-size crucible. The furnaces are operated for a period of 12 hours per day on coke which must be clean, hard, and low in sulphur. In the morning the fire is kindled on the grates and a layer of coke placed on top. The crucible is set on the fuel bed and a mixture of coarse and fine coke packed between the outside of the crucible and the furnace lining. When the crucible reaches a red heat, a ring made from the upper half of a discarded crucible is placed on top of the crucible in order

[1] These crucibles were made by the Vesuvius Crucible Co., of Swissvale, Pa., through the kindness of Mr. F. L. Arensberg, President, to whom the Bureau of Mines is greatly indebted for his interest and generous coöperation.

to prevent notching and the charge is then introduced. Usually it requires from 90 to 120 minutes to melt and heat the charge to the desired pouring temperature.

When the metal is ready to pour, the crucible is removed, by well fashioned tongs of the grab type operated by a chain block, and lowered into a shank and carried to the molds and poured. After pouring, the crucible is immediately returned to the furnace for recharging. The operation requires about six minutes and during that time the crucible cools down to a dull red.

Foundry No. 2 operates sixty-four natural-draft, coke-fired pit furnaces on No. 80-size crucibles. The operations are carried out in a similar manner to those of foundry No. 1, except that no protecting ring is used and the tops of the crucibles are considerably worn down during charging. In foundry No. 1 as much care in handling the crucibles was exercised as maximum production would permit. Rough handling seemed to be the rule in foundry No. 2 and the crucibles were used with what might be called lavishness.

The gripping portion of the tongs was narrow and when undue pressure was applied cut into the crucible and was responsible for shortening its life. After the crucible was carried to the molds in the tongs and poured, it was loosened from the tongs and dragged back across the floor and placed in the furnace for recharging.

Of the crucibles tested, the first two series contained Ceylon and Canadian graphites, and the third and fourth series contained Ceylon and Alabama graphites. In curtailing imports on foreign graphite in order to save ship tonnage, the crucible manufacturers were restricted to the use of 25 per cent American graphite. The following tests were made in order to obtain data relative to the effect upon the life of the crucible of substituting flake graphite for the Ceylon graphite.

Series 1.

The first series consisted of seven members marked Nos. 50 to 56 inclusive. Nos. 50, 51, 52 and 53 represent mixtures of Ceylon and Canadian graphites up to the 25 per cent require-

ments. Nos. 54, 55 and 56 contained 25 per cent Canadian graphite but with variable clay content. The compositions of the crucible bodies are given in Table 1.

TABLE 1.—COMPOSITIONS, SERIES 1.

Mark.	No. 50.	No. 51.	No. 52.	No. 53.	No. 54.	No. 55.	No. 56.
Ceylon graphite	55.0	49.5	45.4	41.3	37.5	37.5	37.5 per cent
Canadian graphite	..	5.5	9.6	13.7	12.5	12.5	12.5 per cent
Dorset ball clay	35.0	35.0	35.0	35.0	35.0	37.5	40.0 per cent
Pot shell	10.0	10.0	10.0	10.0	10.0	10.0	10.0 per cent
Georgia kaolin	..	..	..	..	5.0	2.5	.. per cent
[1]Per cent Ceylon graphite	100	90	82½	75	75	75	75 per cent
Per cent Canadian graphite	0	10	17½	25	25	25	25 per cent

Ten crucibles of No. 70 size were made from each body and shipped to foundry No. 1 for tests. The crucibles were over a week in transit arriving at the foundry November 17th. They were immediately placed in the drier which is heated by steam coils and operated at a temperature of 100° to 110° C.

On December 2nd, two representatives of the Ceramic Station arrived at the foundry to conduct the tests. The crucibles therefore had received 15 days' drying when the tests were started.

The foundry was making several different alloys. Of these, two were produced in greatest quantities. These were called brass No. 3 and brass M1 and were selected as the brasses on which the crucibles were tested. Their compositions are given in Table 2.

TABLE 2.—BRASS COMPOSITIONS.

	No. 3. Per cent.	M1. Per cent.
Copper	78.00	71.00–73.00
Zinc	12.00	19.25–21.25
Lead	7.00	5.50– 6.50
Tin	3.00	1.50– 2.50

[1] Per cent graphite is reported on a basis of 100 parts graphite in the mix. The graphite was screened between 14 and 100 mesh.

The No. 3 brass requires a higher temperature for melting and a longer period of heating. "Rilton's Brass Cleaner" is introduced with the charge of "M1" and carbonate of soda with No. 3. The composition of Rilton's brass cleaner is not given. Its corrosive action on the crucible was less severe than the carbonate of soda, which attacked the crucibles vigorously and seemed to dissolve the clay bond and loosen the graphite grains.

Crucibles not failing through cracking, pinholing, breaking or scalping, gradually wasted away from the insides until the walls became so thin that it would be dangerous to use them again. Such crucibles generally tested high and were classed by the foundry superintendent as "freaks."

The data on the tests for the ten crucibles of each composition are given in Tables 3 to 9 and the average results are compared in Table 10.

The results of the tests are comparatively consistent on crucibles of No. 50 composition as shown in Table 3. This is especially noticeable in the tests on No. 3 brass which show an improve-

TABLE 3.—TESTS ON NO. 50 COMPOSITION—CRUCIBLE SIZE NO. 70.

Wt. lbs., crucible.	No. of furnace.	Average minutes per heat.	No. days in drier.	Kind of brass.	No. of heats.	Cause of failure.
33	25	103	15	No. 3	11	Pinholing
33	3	100	17	No. 3	11	Pinholing
34	22	119	19	No. 3	11	Cracked around crucible
36	23	104	22	No. 3	12	Large hole below bulge
35	7	97	22	No. 3	16	Cracked around crucible
34	25	123	29	No. 3	18	Pinholing
34	31	87	24	M1	34	Pinholing
33	26	133	25	M1	36	Vertical crack at rim
34	28	93	26	M1	30	Vertical crack at rim
32	9	83	30	M1	35	Vertical crack at rim

Averages.	Wt. crucible, lbs.	Minutes per heat.	No. days in drier.	No. of heats.
On No. 3 brass (6 crucibles)	34.2	107.7	20.67	13.17
On M1 brass (4 crucibles)	33.2	99.0	26.25	33.75
On No. 3 and M1	33.7	103.3	23.46	23.46

TABLE 4.—TESTS ON NO. 51 COMPOSITION—CRUCIBLE SIZE NO. 70.

Wt. lbs., crucible.	No. of furnace.	Average minutes, per heat.	No. days in drier.	Kind of brass.	No. of heats.	Cause of failure.
32.5	19	118	15	No. 3	8	Cracked around crucible
34.0	23	106	15	No. 3	9	Cracked around crucible
33.0	25	118	18	No. 3	13	Cracked around crucible
33.0	4	80	19	No. 3	9	Cracked around crucible
32.0	13	90	16	M1	26	Pinholing
32.5	27	147	16	M1	31	Vertical cracks at rim
34.0	9	96	16	M1	32	Bottom broken out
32.0	12	97	16	M1	35	Pinholing
33.0	29	92	16	M1	36	Worn too thin
32.5	26	128	16	M1	39	Worn too thin

Averages.	Wt. crucible, lbs.	Min. per heat.	No. days in drier.	No. of heats.
On No. 3 brass (4 crucibles)	33.1	105.5	16.75	9.75
On M1 brass (6 crucibles)	32.7	108.3	16.00	33.17
On No. 3 and M1	32.9	106.9	16.37	21.46

TABLE 5.—TESTS ON NO. 52 COMPOSITION—CRUCIBLE SIZE NO. 70.

Wt. lbs., crucible.	No. of furnace.	Average minutes, per heat.	No. days in drier.	Kind of brass.	No. of heats.	Cause of failure.
37.0	7	76	15	No. 3	1[1]	Scalping
39.0	22	109	15	No. 3	15	Hole below bulge
37.5	5	90	17	No. 3	9	Pinholing
37.0	3	99	18	No. 3	9	Vertical cracks
35.0	2	101	19	No. 3	3	Vertical cracks at rim
38.0	24	126	22	No. 3	16	Vertical cracks
38.0	24	137	29	No. 3	15	Horizontal crack below bulge
38.0	11	102	19	M1	25	Vertical cracks
39.0	12	96	23	M1	35	Bottom broken out
38.0	30	106	27	M1	1[1]	Scalping at bottom

Averages.	Wt. crucible, lbs.	Min. per heat.	No. days in drier.	No. of heats.
On Brass No. 3 (6 crucibles)	37.4	110.3	20.0	11.17
On brass M1 (2 crucibles)	38.5	99.0	21.0	30.0
On No. 3 and M1	37.9	104.6	20.5	20.58

[1] Rejected, too rapid heating.

TABLE 6.—TESTS ON NO. 53 COMPOSITION—CRUCIBLE SIZE NO. 70.

Wt. crucible, lbs.	No. of furnace.	Average minutes per heat.	No. days in drier.	Kind of brass.	No. of heats.	Cause of failure.
40.0	24	147	18	No. 3	1[1]	Scalping (laminated)
37.0	15	103	19	No. 3	9	Hole at bulge
36.0	2	85	20	No. 3	9	Pinholing
36.0	3	93	22	No. 3	7	Broke around crucible
37.0	3	87	23	No. 3	9	Worn too thin
36.0	23	121	29	No. 3	8	Pinholing
38.0	4	92	30	No. 3	11	Vertical crack below bulge
35.0	10	90	16	M1	14	Pinholing
39.0	11	95	16	M1	22	Vertical cracks
36.0	10	113	29	M1	2[1]	Shattered (flawed)

Averages.	Wt. crucible, lbs.	Min. per heat.	No. days in drier.	No. of heats.
On brass No. 3 (6 crucibles)	36.7	97.3	23.83	8.83
On brass M1 (2 crucibles)	37.0	92.5	16.00	18.00
On No. 3 and M1	36.8	94.9	19.91	13.41

TABLE 7.—TESTS ON NO. 54 COMPOSITION—CRUCIBLE SIZE NO. 70.

Wt. crucible, lbs.	No. of furnace.	Average minutes per heat.	No. days in drier.	Kind of brass.	No. of heats.	Cause of failure.
34.0	23	120	17	No. 3	5	Vertical crack, pinholed
34.0	5	103	18	No. 3	4	Vertical crack at rim
35.0	6	107	18	No. 3	5	Pinholing
35.0	4	99	22	No. 3	8	Cracked around crucible
35.0	8	86	23	No. 3	3	Vertical cracks
36.0	4	97	29	No. 3	8	Horizontal cracks at bulge
35.0	3	92	30	No. 3	7	Vertical cracks below bulge
33.5	15	93	16	M1	22	Vertical cracks at rim
34.5	28	90	16	M1	24	Cracked around bulge
35.0	6	86	19	M1	8	Vertical cracks

Averages.	Wt. crucible, lbs.	Min. per heat.	No. days in drier.	No. of heats.
On brass No. 3 (7 crucibles)	34.9	100.6	22.73	5.71
On brass M1 (3 crucibles)	34.3	89.7	17.0	18.00
On brass No. 3 and M1	34.6	95.1	19.86	11.85

[1] Rejected.

TABLE 8.—TESTS ON NO. 55 COMPOSITION—CRUCIBLE SIZE NO. 70.

Wt. crucible, lbs.	No. of furnace.	Average minutes per heat.	No. of days in drier.	Kind of brass.	No. of heats.	Cause of failure.
34.5	2	101.0	15	No. 3	5	Pinholing
35.0	15	120.5	16	No. 3	9	Pinholing
35.0	2	89.0	16	No. 3	10	Cracked around crucible
34.0	2	93.0	17	No. 3	10	Cracked around crucible
35.0	5	91.0	19	No. 3	13	Pinholing
34.0	3	91.5	29	No. 3	8	Vertical crack below bulge
34.0	5	116.5	30	No. 3	5	Vertical cracks
35.0	10	93.0	18	M1	13	Pinholing
35.0	28	86.0	24	M1	15	Pinholing
36.0	29	90.8	26	M1	27	Vertical crack near rim

Averages.	Wt. crucible, lbs.	Min. per heat.	No. days in drier.	No. of heats.
On brass No. 3 (7 crucibles)	34.5	97.8	20.28	8.57
On Brass M1 (3 crucibles)	35.3	89.7	22.67	18.33
On No. 3 and M1	34.9	93.7	21.47	13.45

TABLE 9.—TESTS ON NO. 56 COMPOSITION—CRUCIBLE SIZE NO. 70.

Wt. crucible, lbs.	No. of furnace.	Average minutes per heat.	No. of days in drier.	Kind of brass.	No. of heats.	Cause of failure.
35.0	3	92.0	15	No. 3	7	Pinholing
34.0	18	100.0	15	No. 3	12	Pinholing
35.0	3	100.5	16	No. 3	3	Vertical cracks
35.0	6	90.0	17	No. 3	5	Vertical cracks
35.0	23	108.0	18	No. 3	8	Pinholing
34.0	6	106.3	22	No. 3	11	Pinholing
35.0	2	95.0	29	No. 3	8	Vertical crack below bulge
33.5	30	79.3	16	M1	23	Pinholing
34.0	31	89.6	16	M1	25	Hole below bulge
35.0	14	85.0	17	M1	28	Bottom broke on drawing

Averages.	Wt. crucible, lbs.	Min. per heat.	No. days in drier.	No. of heats.
On brass No. 3 (7 crucibles)	34.7	98.8	18.86	7.71
On brass M1 (3 crucibles)	34.2	84.6	16.33	25.33
On No. 3 and M1	34.4	91.7	17.60	16.52

TABLE 10.—SUMMARY OF TESTS ON SERIES 1.

Mark.	Average minutes per heat.			Average days in drier.			Average number of heats.		
	On No. 3 brass.	On M1 brass.	Av. on No. 3 and M1.	On No. 3 brass.	On M1 brass.	Av. on No. 3 and M1.	On No. 3 brass.	On M1 brass.	Av. on No. 3 and M1.
50	107.7	99.0	103.3	20.67	26.25	23.46	13.17	33.75	23.46
51	105.5	108.3	106.9	16.75	16.00	16.37	9.75	33.17	21.46
52	110.3	99.0	104.6	20.00	21.00	20.50	11.17	30.00	20.58
53	97.3	92.5	94.9	23.83	16.00	19.91	8.83	18.00	13.41
54	100.6	89.7	95.1	22.73	17.00	19.86	5.71	18.00	11.85
55	97.8	89.7	93.7	20.28	22.67	21.47	8.57	18.33	13.45
56	98.8	84.6	91.7	18.86	16.33	17.60	7.71	25.33	16.52

ment in the life of the crucible with increase in the length of time of drying. In general this characteristic is shown in the other tests but is not borne out as consistently. Neither the weight of the crucible nor the length of time of heating seem to bear any relation to the crucible's life. However, the rate of heating is important since too rapid heating may cause cracking or scalping. According to Gillett[1] "Allowing the crucible to remain in the fire longer than is necessary; that is, not taking the pot out when the metal is ready, or allowing the metal to 'soak,' increases the wear on the crucible."

In order to compare the serviceability of the crucibles the life of the crucible is expressed in per cent (Table 11) on a basis of 100 for the crucible giving highest service on each brass and on the average for both brasses.

TABLE 11.—PER CENT LIFE OF CRUCIBLES.

Mark.	On No. 3 brass.	On M1 brass.	Av. on No. 3 and M1.
50	100.00	100.00	100.00
51	74.03	98.28	91.47
52	84.05	88.89	87.72
53	67.05	53.33	57.16
54	43.36	53.33	50.51
55	65.07	54.31	57.33
56	58.54	75.05	70.42

[1] Loc. cit., 165.

The replacement of Ceylon graphite by the Canadian graphite has decreased the life of the crucible up to a replacement of 25 per cent, which has evidently lowered the service of the crucible by nearly 33 per cent on brass No. 3, $46^2/_3$ per cent on brass M1, and nearly 43 per cent on the average for both brasses.

In Nos. 54, 55 and 56, the ratio of Ceylon to Canadian graphite remains constant at 75 per cent to 25 per cent. As compared to No. 53, five parts of the graphite are replaced by clay. In No. 54 the replacement is Georgia kaolin and has evidently resulted in lowering the life of the crucible.

Georgia kaolin is very refractory and is a clay of low bonding strength as compared to bonds of the ball clay type. At brass melting temperatures, Georgia kaolin remains too porous and friable to impart strength and resistance to slagging action. However, at steel melting temperatures, the use of a small amount of Georgia kaolin in the crucible would in all probability be beneficial.

In No. 55 as compared to No. 54, $2^1/_2$ parts Georgia kaolin is replaced by Dorset ball clay. The replacement has improved the life of the crucible and has resulted in a crucible giving equally as good service as No. 53. Evidently the good effects of the ball clay have balanced the ill effects of the kaolin.

A comparison of No. 56 with No. 53 shows an improvement in the life of the crucible by the replacement of 5 parts of graphite by 5 parts Dorset ball clay as against a decrease in service by an equal substitution of kaolin for graphite (No. 54).

The Ceylon graphite has a granular or splintery form with a comparatively low surface factor and low bulkiness, while the Canadian grain is thin, plate-like in form, of high surface factor and higher bulkiness. Therefore, more clay is necessary to bond the flake graphite than is necessary for bonding the Ceylon graphite. The average results indicate an improvement in the life of the crucible by an increase of 5 parts ball clay at the expense of graphite.

Series 2.

Series 2 consisted of two members, Nos. 58 and 59. Fifteen crucibles of each of No. 80 D-size were tested. The graphite

in No. 58 was Canadian flake and that in No. 59 a mixture of 75 per cent Ceylon and 25 per cent Canadian flake. Compositions of the crucibles are given in Table 12.

TABLE 12.

Mark.	No. 58.	No. 59.
Ceylon graphite	0.0 per cent	41.25 per cent
Canadian graphite	55.00 per cent	13.75 per cent
Dorset ball clay	35.00 per cent	35.00 per cent
Pot shell	5.00 per cent	5.00 per cent
Sand	5.00 per cent	5.00 per cent

The crucibles were tested at foundry No. 2. The drier at this foundry is operated at a temperature varying from 90° to 110° C. The heat is supplied by hot air from a "stove" outside of the drying room. After the crucibles had received $3^1/_2$ days' drying treatment, the tests were made. It was realized that the short

TABLE 13.—TESTS OF NO. 58 COMPOSITION—CRUCIBLE SIZE NO. 80 D.

No. of furnace.	Days in drier.	Av. minutes per heat.	No. of heats.	Cause of failure.
3	3.5	55	13	Bottom cracked, careless handling
4	3.5	55	13	Bottom cracked, careless handling
1	4.5	55	13	Large hole in side
2	4.5	55	13	Horizontal crack at middle
3	5.5	55	7	Broken in half on removal, 8th heat
4	5.5	55	13	Pinholing
6	5.5	55	15	Vertical crack at rim
8	5.5	55	19	Horizontal crack at middle
1	5.5	55	12	Broke on removal from furnace
2	5.5	55	12	Bottom broke on charging
4	5.5	55	11	Large hole in side
5	5.5	55	12	Pinholing
6	5.5	55	6	Vertical crack at rim
7	5.5	55	11	Pinholing
1	8.5	55	12	Pinholing

Total heats, 15 crucibles..... 182.
Average heats per crucible..... 12.13
Graphite—100 per cent Canadian.

drying period was insufficient and would be detrimental to the life of the crucibles, but owing to lack of time, it was considered that comparable results would be obtained from the tests on 15 crucibles of each composition.

The tests were made on brass composed of 70 per cent copper and 30 per cent zinc. About half the charge was scrap and half new metal. After the charge had melted and about 15 minutes before removing the crucibles for pouring, a hand-full of salt and a shovel-full of charcoal were thrown on top of the metal. The results of the tests are given in Tables 13 and 14.

TABLE 14.—TESTS OF NO. 59 COMPOSITION—CRUCIBLE SIZE NO. 80 D.

No. of furnace.	Days in drier.	Av. minutes per heat.	No. of heats.	Cause of failure.
1	3.5	55	13	Pinholing
2	3.5	55	20	Top worn down
3	4.5	55	13	Pinholing
4	4.5	55	13	Pinholing
1	5.5	55	18	Vertical crack at middle
2	5.5	55	11	Vertical crack at middle
5	5.5	55	12	Vertical crack at middle
7	5.5	55	15	Vertical crack below bulge
3	5.5	55	22	Pinholing
4	5.5	55	5	Broken by tongs in placing
7	5.5	55	16	Pinholing
1	5.5	55	4	Horizontal crack, squeezed by tongs
2	5.5	55	7	Horizontal crack, squeezed by tongs
3	5.5	55	9	Horizontal crack at middle
3	8.5	55	11	Broken around bulge

Total number of heats, 15 crucibles..... 189.00
Average number of heats per crucible..... 12.60
Graphite, 75 per cent Ceylon and 25 per cent Canadian.

The difference in the lives of the Nos. 58 and 59 crucibles was small, having been less than $^1/_2$ heat per crucible. The comparison of service on a percentage basis is; No. 58, 96.27 per cent; and No. 59, 100 per cent.

Series A and B.

Series A was made in order to observe the effect of replacing Ceylon graphite by Alabama No. 1 crucible flake graphite. In

series B, "coked" Alabama graphite replaced the Ceylon. In order to change the flake form of graphite to the granular form, a mixture of three parts Alabama flake graphite, between 20 and 100 mesh size, and 1 part dust passing the 100 mesh sieve was briquetted with 15 per cent tar under a pressure of 5000 pounds per square inch, coked at 1000° C, crushed, screened through 16 mesh and passed over a 100 mesh screen to remove the surplus fines.

The Ceylon graphite was graded so as to produce the same screen analysis as the coked Alabama. The Alabama crucible flake, which all passed through the 20 mesh screen, was used as received from the dealer. The screen analyses are given in Table 15 and the compositions of series A and B in Tables 16 and 17.

Four crucibles of each member of the A and B series were made in No. 60 size and were originally intended for tests in a brass foundry at Columbus, Ohio, but owing to the fact that the output of the foundry was small, and that no adequate drier was

TABLE 15.

	Coked Alabama.	Ceylon.	Alabama flake.
Between 16 and 20 mesh	16.0	16.0	0.0
Between 20 and 40 mesh	25.0	25.0	11.0
Between 40 and 100 mesh	55.0	55.0	85.5
Through 100 mesh	4.0	4.0	3.5

TABLE 16.—COMPOSITIONS, SERIES A.

Mark.	A—1.	A—2.	A—3.	A—4.	A—5.
Ceylon graphite	55.0	42.5	30.0	17.5	0.0
Alabama graphite	0.0	12.5	25.0	37.5	53.0
Dorset ball clay	20.0	20.0	20.0	20.0	20.0
McLendon bond clay	10.0	10.0	10.0	10.0	10.0
Kentucky ball clay	5.0	5.0	5.0	5.0	5.0
Illinois kaolin	5.0	5.0	5.0	5.0	7.0
Glass sand	5.0	5.0	5.0	5.0	5.0
Per cent Alabama graphite	0	22.73	45.45	68.18	100
Per cent Ceylon graphite	100	77.27	54.55	31.82	0

TABLE 17.—COMPOSITIONS, SERIES B.

Mark.	B—1.	B—2.	B—3.	B—4.
Ceylon graphite	42.5	30.0	17.5	0.0
Coked Alabama graphite	12.5	25.0	37.5	53.0
Dorset ball clay	20.0	20.0	20.0	20.0
McLendon bond clay	10.0	10.0	10.0	10.0
Kentucky ball clay	5.0	5.0	5.0	5.00
Illinois kaolin	5.0	5.0	5.0	7.0
Glass sand	5.0	5.0	5.0	5.0
Per cent coked Alabama graphite	22.73	45.45	68.18	100
Per cent Ceylon graphite	77.27	54.55	31.82	0

TABLE 18.—SERIES A, CRUCIBLE SIZE NO. 60.

Mark.	Wt. crucible, lbs.	Days in drier.	No. of furnace.	Kind of brass.	Average minutes per heat.	No. of heats.	Cause of failure.
A1	30.0	19	2	No. 3	92.50	5	Vertical cracks below bulge
A1	31.0	23	4	No. 3	91.00	9	Vertical cracks below bulge
A1	31.0	20	10	M1	94.00	10	Pinholing
A1	30.0	22	31	M1	124.00	7	Pinholing
A2	31.0	17	8	No. 3	86.80	7	Vertical cracks
A2	30.5	18	22	No. 3	110.00	9	Horizontal crack at bulge
A2	28.0	23	29	M1	92.67	15	Pinholing
A2	30.0	26	10	M1	74.00	8	Pinholing
A3	30.0	15	4	No. 3	89.00	10	Vertical cracks at bulge
A3	30.0	17	15	No. 3	99.00	13	Vertical cracks
A3	30.0	19	5	M1	86.43	27	Pinholing at bottom
A3	29.0	29	30	M1	86.33	21	Worn too thin
A4	30.0	18	4	No. 3	94.00	8	Cracked diagonally at bulge
A4	29.0	19	23	No. 3	112.00	9	Horizontal crack at bottom
A4	28.0	19	28	M1	109.40	20	Pinholing
A4	29.0	20	13	M1	96.00	20	Vertical cracks
A5	28.0	22	15	No. 3	103.00	12	Pinholing
A5	27.0	22	25	No. 3	120.00	15	Cracked around crucible
A5	28.0	25	11	M1	96.33	29	Vertical crack near bottom
A5	26.0	29	10	M1	84.60	28	Vertical crack at bulge

available, a more suitable place was sought for the testing. A communication with the superintendent of foundry No. 1 brought

forth the reply that he could use the crucibles, although their equipment was designed to handle No. 70 size.

The A and B series were shipped to foundry No. 1 with the crucibles of Series 1 and tested at the same time. The A and B series were placed in the drier at the same time as Series 1 and therefore received 15 days' drying before the tests were begun.

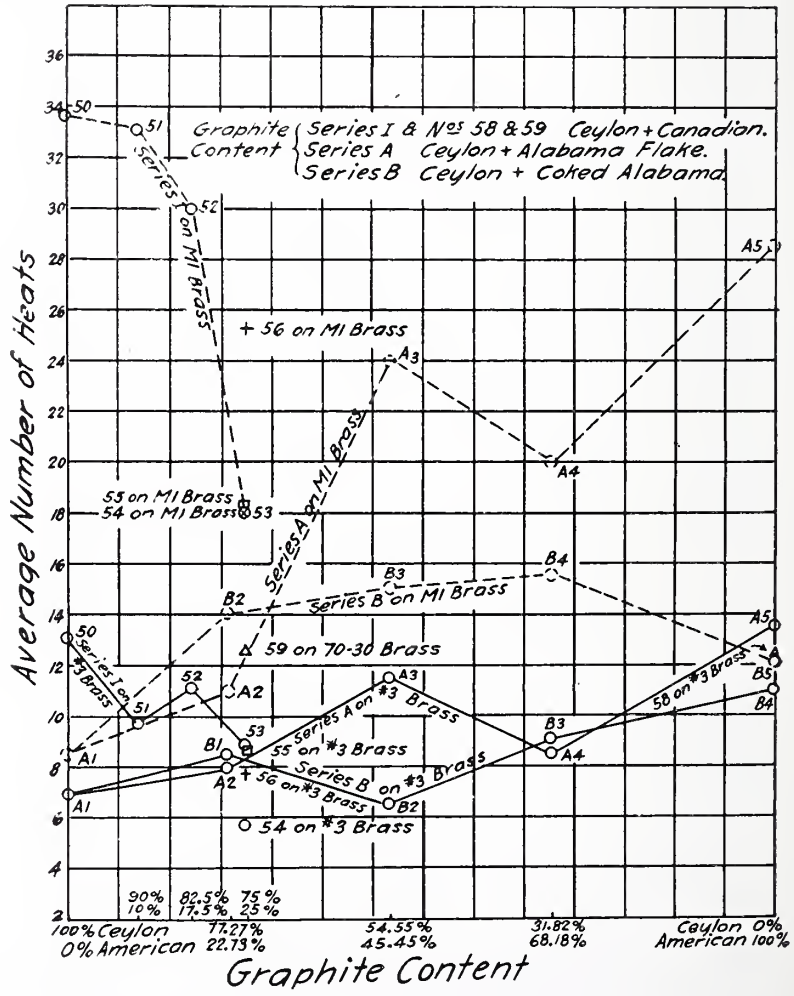


FIG. 1.—Average number of heats on different brasses.

During the tests it was observed, by the two representatives conducting the tests, that the equipment of the foundry was not suited to the No. 60 size crucibles. This was especially noticeable in the lifting tongs which were designed to fit the No. 70 size, and the lives of the smaller crucibles were affected by the unequal squeezing of the tongs. Since the tongs were of the grab type, the pressure on the crucible depended upon the weight of the crucible and its charge, so that the squeezing effect was more or less constant for all crucibles of the same size.

The crucibles were tested on both No. 3 and M1 brass. Tests for Series A are recorded in Table 18, Series B in Table 19, and the average tests for both series in Table 20.

The number of crucibles tested in series A and B is not sufficient from which conclusions can be accurately drawn. Owing to the erratic behavior of crucibles in use, at least ten crucibles of each composition should be tested. At the time the crucibles

TABLE 19.—SERIES B, CRUCIBLE SIZE NO. 60.

Mark.	Wt. crucible, lbs.	Days in drier.	No. of furnace.	Kind of brass.	Average minutes per heat.	No. of heats.	Cause of failure.
B1	32.0	20	3	No. 3	90.00	5	Vertical cracks
B1	31.0	23	22	No. 3	107.00	12	Vertical and horizontal cracks
B1	31.5	24	30	M1	89.00	15	Pinholing
B1	31.0	29	13	M1	95.33	13	Pinholing
B2	29.0	17	4	No. 3	102.50	5	Vertical cracks at bulge
B2	30.0	23	8	No. 3	108.33	8	Vertical cracks
B2	29.0	24	23	M1	107.33	9	Vertical cracks
B2	30.0	24	27	M1	132.50	21	Vertical cracks at rim
B3	31.0	20	4	No. 3	85.0	1[1]	Cracked, too rapid heating
B3	29.0	22	5	No. 3	100.00	9	Vertical cracks
B3	31.0	26	14	M1	124.67	15	Vertical cracks below bulge
B3	31.0	30	27	M1	122.50	16	Pinholing
B4	28.0	18	24	No. 3	134.75	14	Cracked around crucible
B4	30.0	23	2	No. 3	99.00	8	Vertical cracks
B4	29.0	20	30	M1	113.67	10	Pinholing
B4	29.0	30	14	M1	91.67	14	Vertical crack at bulge

[1] Rejected.

TABLE 20.—AVERAGE TESTS ON SERIES A AND B.

Mark.	Average minutes per heat.			Average days in drier.			Average number of heats.		
	On brass No. 3.	On brass M1.	Av. on No. 3 and M1.	On brass No. 3.	On brass M1.	Av. on No. 3 and M1.	On brass No. 3.	On brass M1.	Av. on No. 3 and M1.
A1	91.7	109.0	100.4	21.0	21.0	21.0	7.0	8.5	7.75
A2	98.4	83.3	90.8	17.5	24.5	21.0	8.0	11.0	9.75
A3	94.0	86.4	90.2	16.0	24.0	20.0	11.5	24.0	17.75
A4	103.0	102.7	102.8	18.5	19.5	19.0	8.5	20.0	14.25
A5	111.5	90.5	101.0	22.0	27.0	24.5	13.5	28.5	21.00
B1	98.5	92.2	95.3	21.5	26.5	24.0	8.5	14.0	11.25
B2	105.4	119.9	112.6	20.0	24.0	22.0	6.5	15.0	10.75
B3	92.5	123.6	108.0	21.0	28.0	24.5	9.0	15.5	12.25
B4	116.9	102.6	109.7	20.5	25.0	22.7	11.0	12.0	11.50

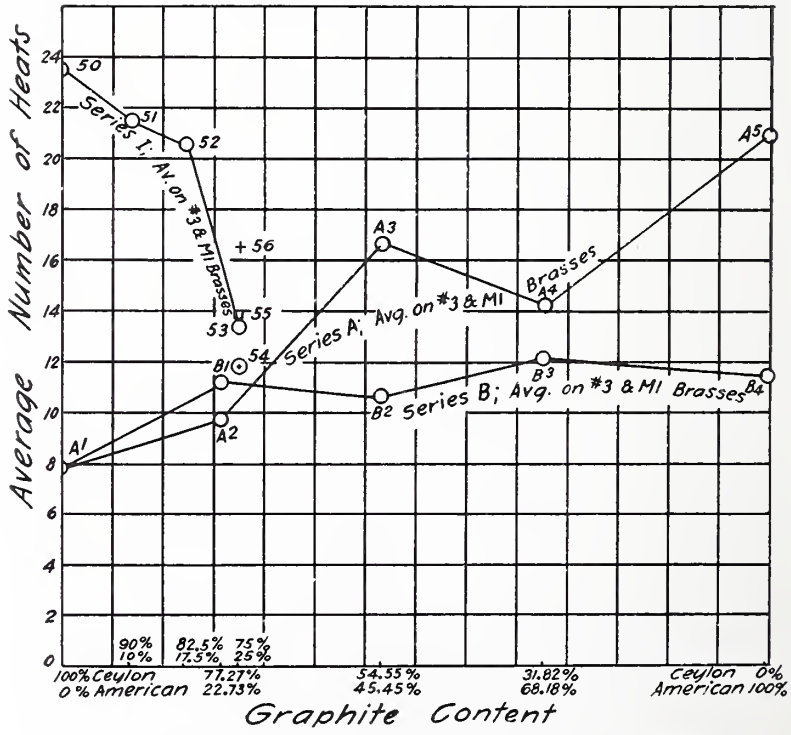


FIG. 2.—Number of heats averaged on Nos. 3 & M1 brasses.

were made, the materials on hand were limited, which limited the number of crucibles to four for each composition.

The average results for the crucibles tested on the different brasses are plotted in Fig. 1 and the average results of the tests on No. 3 and M1 brasses are shown graphically in Fig. 2.

In Fig. 1 the curves showing the behavior of Series A on both brasses show considerable similarity. The curves take the same general direction, rising and falling at the same corresponding points, but this similarity is lacking in the other series.

Conclusions.

In so far as the work has progressed, it shows that the substitution of Canadian graphite up to 25 per cent for Ceylon, decreased the life of the crucible (Series 1). Crucibles containing 100 per cent Canadian graphite gave nearly as good service as those containing 25 per cent Canadian and 75 per cent Ceylon (Series 2). The substitution of Alabama flake graphite for the Ceylon improved the crucible's life (Series A). The substitution of coked Alabama graphite for the Ceylon improved the life of the crucible (Series B). The substitution of the coked Alabama graphite for the Alabama flake up to 22.73 per cent increased and higher substitutions decreased the life of the crucible (Series A and B, Fig. 2). No material advantage was gained by converting the Alabama flake graphite to the granular form.

In crucibles containing 75 per cent Ceylon and 25 per cent Canadian graphite, the increase in clay content by Georgia kaolin lowered the life of the crucible. Replacement of the kaolin by Dorset ball clay improved the life of the crucible (Nos. 53, 54, 55 and 56, Fig. 2).

The marked difference in the service of A1 and No. 50 is noteworthy. Both contain all Ceylon graphite. A1 was smaller by three pints capacity than No. 50. The smaller the crucible the greater the number of heats it should stand, according to the curves shown by Gillett;[1] in which the service of the crucible, expressed in number of heats, is plotted against the maker's number. A No. 60 size crucible should stand approximately $4^1/_4$ per cent more heats than a No. 70 size. This appears to be

[1] Loc. cit., p. 159.

the only advantage that A1 might have over No. 50. The advantage is small and is more than offset by the disadvantages.

The larger the crucible the larger should be the graphite grains, in order to produce a crucible of maximum strength and service. The graphite in No. 50 was screened between 14 and 100 mesh, whereas, the graphite in A1 was screened through 16 mesh. The graphite of No. 50 showed considerably more material remaining on the 40 mesh screen than the graphite of A1. In respect to size of grain, No. 50 crucibles had the advantage. Not only was A1 at a disadvantage with respect to size of grain, but its life was also materially affected by unequal squeezing of the ill-fitting tongs.[1]

Crucibles giving highest average service on M1 brass and on the averages for both No. 3 and M1, were those containing all Ceylon graphite (No. 50), whereas, the crucible giving highest service on No. 3 brass was A5 in which the graphite was all Alabama flake. It therefore appears that the Alabama graphite offers promising possibilities for the manufacture of graphite crucibles.

When the refining process is completed the No. 1 Alabama flake seldom exceeds 20 mesh in size. Its crucible-making value would therefore be limited by the size of the crucible—it being doubtful whether it could be used successfully for the manufacture of the larger sizes. By the judicious use of sand and pot-shell, to overcome the slipperiness and tendency to laminate, it is probable that crucibles giving high service can be made from Alabama graphite up to a No. 70 size or even larger.

Bureau of Mines,
Mining Experiment Station,
Columbus, Ohio.

[1] See Gillett, Loc. cit., pp. 161–163.

IMPACT TESTS AND POROSITY DETERMINATIONS ON SOME AMERICAN HOTEL CHINA AND SEMI-PORCELAIN PLATES.[1]

By Homer F. Staley and J. S. Hromatko, Washington, D. C.

Impact Tests.

Determination of the physical properties of chinas and semi-porcelains offer no particular difficulties when test pieces suitable for use in the various kinds of apparatus ordinarily employed in testing laboratories can be made to order. It is then possible to determine with a fair degree of accuracy the cross-bending strength, the crushing strength, modulus of rupture or any other mechanical characteristic desired.

Conditions are altogether different when it comes to making tests for determining the serviceability of manufactured wares such as hotel chinas. In the first place the forms of the pieces available for testing are not suited for use in most testing machines. Moreover, the variety of ways in which a piece may fail in service makes it necessary to determine just what properties are to be studied and to actually correlate the tests conducted with service conditions.

Hotel table wares fail in three ways, by marring of the glaze, by chipping, and by breaking. The marring of the glaze by cutlery marks may be due to chemical instability of the glaze, as pointed out by Ashley,[2] or to abrasion. A combination of chemical tests and some form of light impact or abrasion test would seem to be appropriate for determining the liability of glazes to fail in this manner.

Chipping is the result of blows too light to cause cracking of the ware. For determining resistance to failure in this way an impact test of moderate intensity should be satisfactory. A true impact machine may be used or some form of rattler. The

[1] By permission of the Director, Bureau of Standards.

[2] *Trans. Am. Ceram. Soc.*, 13, 226 (1911).

latter machines give a combination of impact and rubbing actions. In a variety of forms they have been used by Watts,[1] Ellsworth Ogden,[2] Lester Ogden,[3] and others for determining the resistance to abrasion of porcelain samples. The use of rattler machines for testing plates and similar articles of table ware is attended by difficulties. The pieces are too large to be used whole, and if they are broken up, the presence of raw edges and sharp corners introduces factors not operative in actual service.

Breaking of table wares in service is in the great majority of cases due to severe impact caused by the dropping of the piece. A true impact test seems to be indicated for determining the resistance of wares to failure in this way. The impact necessary to break a piece of ware and the type of apparatus to be used in testing depend, among various things, on the shape and size of the piece. In this work, seven-inch plates, which were actually about nine inches in diameter, were used as samples.

When a plate is dropped it may fall face downward, on edge, or with the foot downward, and the probability of breaking will vary with the manner in which it falls. Experience has proven that they seldom fall face downward, often fall on edge, but most often fall foot downward. This is no doubt due to the shape of the piece and the weight of the foot. When a plate falls foot downward, the probabilities are against it falling perfectly flat on to a perfectly flat surface. When such cases do occur, the force of the impact is distributed through the whole foot, and the method of failure is different from that in the more common case where one part of the foot strikes first and takes the force of the blow.

The manner in which a broken plate fell may often be determined by study of the location of the fractures. Fig. 1 shows the fracture characteristic of a plate broken by being dropped on edge. Fig. 2 shows the breaking in an irregular manner into many small pieces of a plate dropped foot downward squarely onto a smooth, flat surface. When one point of the foot of the plate strikes first, the result is a V-shaped break. In Fig. 3

[1] *Trans. Am. Ceram. Soc.*, **4**, 107 (1901 2).
[2] *Ibid.*, **7**, 370 (1905).
[3] *Ibid.*, **13**, 395 (1911-2).

we see three lines of fracture radiating from the point of the foot on which a plate falling in this manner struck. Of course these typical fractures are not invariably shown. It is very probable

FIG. 1.

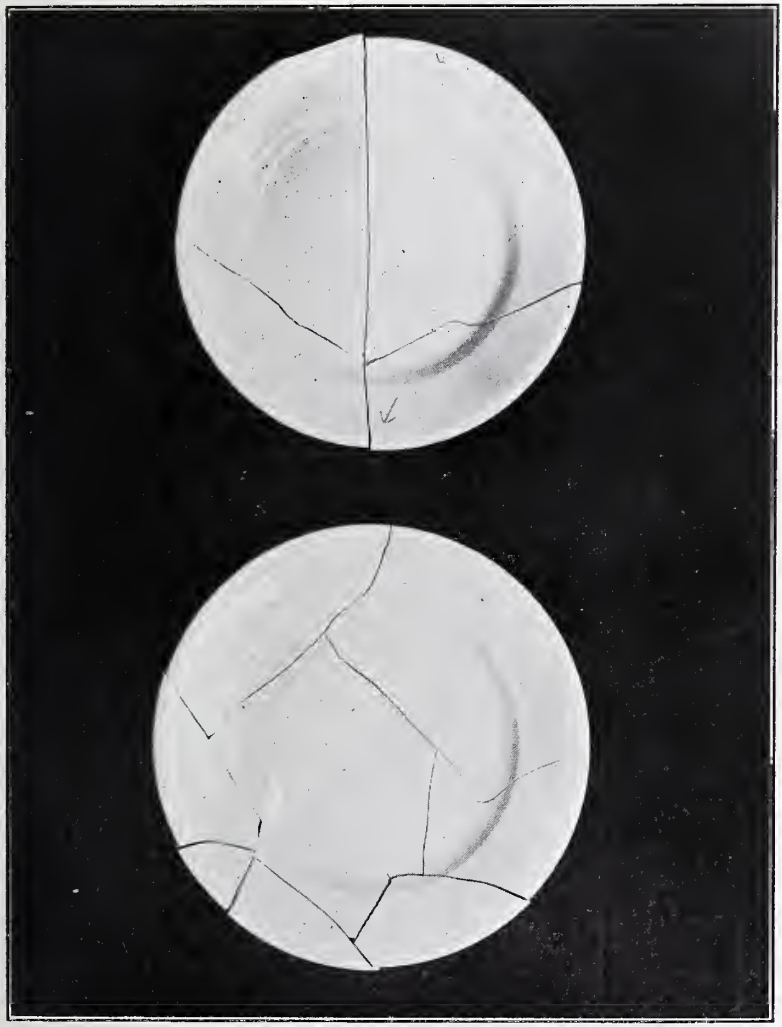


FIG. 2.

that Figs. 1 and 3 show simply variations of the same general type of failure, for sometimes a plate falling on edge may have the kind of fracture shown in Fig. 3 and one falling on one point

FIG. 3.

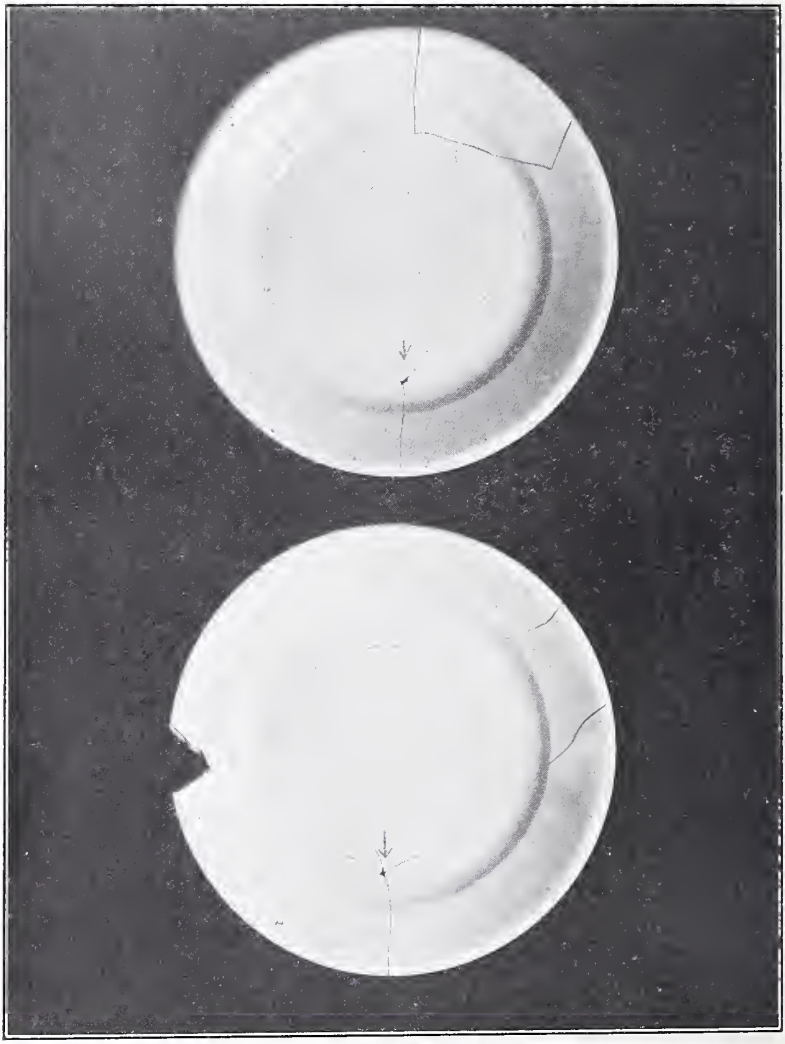


FIG. 4.

of the foot may have the kind of break shown in Fig. 1. This would indicate that failure of the foot at one point is the controlling factor when a plate breaks from falling either onto its edge or

FIG. 5.

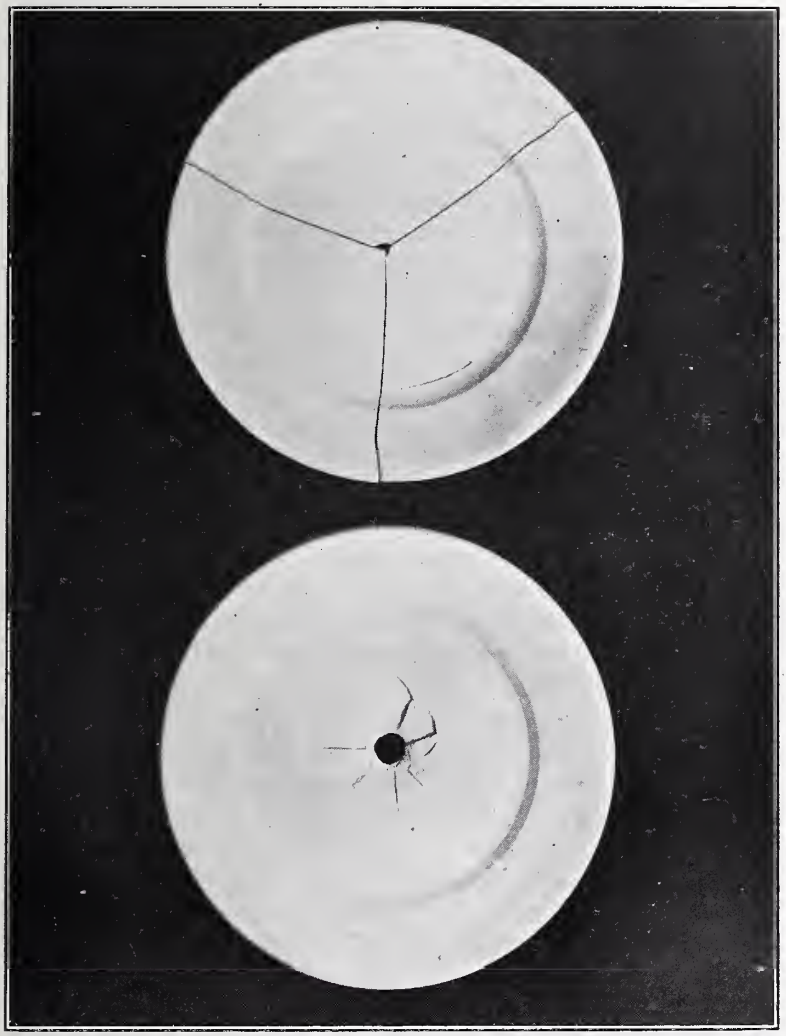


FIG. 6.

one point of its foot. That the impact tests used in this work approximated the conditions obtaining when a plate falls onto one point of its foot is shown by Fig. 4, which is characteristic of the majority of the breaks obtained in these tests.

In the preliminary work incident to these tests two attempts were made to employ impact tests in which the point of impact was at the center of the plates. In one method, the plates were bedded solidly on the foot and the impact blow was delivered in the center of the face. Three lines of fracture radiating from the center of the piece, as shown in Fig. 5, were obtained with every plate tested. In the other method, the plates were bedded on a ring just fitting into the bowl and the blow was delivered onto the center of the bottom. In these cases failure was characterized by crushing of the material immediately beneath the point of impact and by the breaking out of a cone shaped section on the opposite side of the plate. The action was the same as if a blunt punch had been driven through the plate. This type of failure is illustrated in Fig. 6. It is interesting to note that with plates of the same make in the first method of testing the average energy of impact necessary to produce fracture was 0.45 foot-pounds, and in the second was 1.44 foot pounds.

It was finally decided to test plates by impact on one spot on the foot. This test is supposed to approximate the conditions when a plate is broken by being dropped onto one spot on its foot which is, as stated above, a very common manner of breaking in service.

The apparatus used for the impact tests was a heavy metal frame for holding the samples and a pendulum consisting of a $1^1/_4$ pound flattened steel ball at the end of a 24-inch thin and stiff wire arm. The hammer had a spherical striking surface with a radius of $1^1/_8$ inches. A clear, white pine board, $^5/_8$ inch thick, was placed in the frame and then the plate to be tested was held loosely against this. The various parts of the apparatus were so spaced that with this arrangement the point of impact of the hammer would be on the bottom of the foot of the plate and directly below the point of suspension of the pendulum.

Preliminary tests had shown that clamping the plate solidly

against the bearing board introduced pressure strains not present when a plate is dropped and also influenced the distribution of the shock in such manner that both the energy necessary to produce fracture and the type of fracture were altered. In the course of the tests it was found that some makes of vitrified china plates were warped so badly that the use of a bearing plate accommodating itself more readily to irregularities of structure would have been desirable. Such a bearing plate would probably have given more uniform results in some of the individual lots of plates and higher results in all lots. However, it is not probable that it would have altered the relative positions of the various lots. It is rather difficult to determine the effect of highly resilient bearing plates, and the use of these in testing should be avoided when possible.

In making the tests the pendulum was first allowed to swing through 30 degrees, then 35 degrees, and so on, the angle of swing being increased each time by five degrees until fracture occurred.

The energy of impact of a swinging pendulum, neglecting the effect of friction and the weight of the arm, is given by the simple formula

(1) $$E = HW$$

in which

E = energy of impact in foot-pounds.
H = height of drop in feet.
W = weight of hammer in pounds.

The value of H may be determined by direct measurement or by calculation. When the angle of swing is less than 90 degrees and the point of impact is located, as in this case, perpendicularly below the point of suspension, H is given by the formula

(2) $$H = 1 - \text{cosine angle } A \times L$$

in which

Angle A = angle with vertical made by pendulum arm at time of release;
L = length of arm in feet.

Substitution of this value of H in Formula 1 gives

(3) $$E = 1 - \text{cosine angle } A \times L \times W.$$

The results of the tests were calculated by this formula in all cases in which the angle of swing was less than 90 degrees. Of

course when the angle is 90 degrees, H equals L. If the pendulum arm is sufficiently rigid to prevent bending during the dropping of the hammer, the same apparatus may be used, and H can be calculated for angles of swing greater than 90 degrees by the following formula:

(4). $$H = (1 + \text{sine angle A})\, L$$

This formula was used for the angle of swing of 95 degrees used in one case.

It is impossible to figure the cumulative effect of blows of increasing severity, due to lack of data on the mechanical properties of semi-porcelain and china. The work of O'Connor[1] on impact tests of tiles indicates that the energy of impact necessary to produce fracture in these materials is not materially affected by previous blows not sufficiently severe to produce fracture.

TABLE I.—VALUE OF I — COSINE ANGLE A FOR VARIOUS ANGLES.

Angle in degrees.	Value of 1 — cosine.	Angle in degrees.	Value of 1 — cosine.	Angle in degrees.	Value of 1 — cosine.
5	0.00381	35	0.18085	65	0.57738
10	0.01519	40	0.23396	70	0.65798
15	0.03408	45	0.29289	75	0.74118
20	0.06031	50	0.35721	80	0.82635
25	0.09369	55	0.42642	85	0.91285
30	0.13397	60	0.50000	90	1.00000

The data of the impact tests are shown in Tables 2 and 3. Examination of these shows that the total variation in value of energy of impact in individual groups was quite large. As stated above this may be due in part to the poor bedding of warped plates, which would tend to give low values. However, the values for the different lots of plates fall into distinct groups. The relative rating of the different groups according to average energy of impact is not altered if we select as the basis of rating either the maximum individual value in each group or the average of either the three of four highest values in each group. It is not materially changed if we rate the groups according to the minimum value in each group. Of course, it is realized that a larger number of samples would have been desirable, but these were not available. Since this work was rather a pioneer attempt in the field, and gives results of considerable significance, it is to be expected that it will lead to more comprehensive investigations

[1] *Trans. Am. Ceram. Soc.*, **15**, 232 (1913).

These tests show rather conclusively that the seven lots of heavy hotel-ware plates tested varied distinctly in their ability to withstand impact blows similar to those such wares are subjected to in service. Also, that as far as withstanding heavy impact is concerned the china plates were not superior to the semi-porcelains. It should be noted, however, that the latter chipped with slight blows while the china plates did not chip.

TABLE 2.—DATA OF IMPACT TESTS ON SEVEN-INCH HEAVY SEMI-PORCELAIN HOTEL PLATES.

Factory.	Plate.	Angle of swing in degrees.	Energy of impact, foot-pounds.	Thickness of bottom at thinnest point, in inches.	Energy of impact, per 0.001" thickness, foot-pounds.
A.....	1	55	1.07	0.177	
	2	55	1.07	0.206	
	3	50	0.89	0.188	
	4	45	0.73	0.214	
	5	45	0.73	0.187	
	6	50	0.89	0.243	
	Average.....		0.87	0.202	0.00430
B.....	7	75	1.86	0.251	
	8	65	1.44	0.246	
	9	60	1.25	0.242	
	10	70	1.64	0.252	
	11	60	1.25	0.243	
	12	50	0.89	0.243	
	Average.....		1.37	0.246	0.00558
C.....	13	70	1.64	0.244	
	14	65	1.44	0.241	
	15	70	1.64	0.241	
	16	90	2.50	0.267	
	17	60	1.25	0.240	
	18	95	2.72	0.245	
	Average.....		1.86	0.246	0.00752
D.....	19	60	1.25	0.276	
	20	55	1.07	0.295	
	21	35	0.45*	0.284	
	22	50	0.89	0.276	
	23	55	1.07	0.286	
	24	50	0.89	0.293	
	Average.....		1.03	0.285	0.00357

* Bad break, not included in average.

TABLE 3.—DATA OF IMPACT TESTS OF SEVEN-INCH HEAVY CHINA HOTEL PLATES.

Factory.	Plate.	Angle of swing, degrees.	Energy of impact, foot-pounds.	Thickness at bottom of thinnest point, inches.	Energy of impact per 0.001″ thickness, foot-pounds.
E†	25	45	0.73	0.240	
	26	60	1.25	0.210	
	27	40	0.58*	0.218	
	28	50	0.89	0.222	
	29	50	0.89	0.214	
	30	55	1.07	0.229	
	Average		0.97	0.222	0.00437
F†	31	45	0.73	0.228	
	32	70	1.64	0.227	
	33	45	0.73	0.228	
	35	65	1.44	0.229	
	34	55	1.07	0.229	
	36	55	1.07	0.242	
	Average		1.11	0.230	0.00484
G	37	55	1.07	0.288	
	38	80	2.07	0.276	
	39	60	1.25	0.284	
	40	65	1.44	0.268	
	41	65	1.44	0.288	
	42	70	1.64	0.275	
	Average		1.48	0.280	0.00528

* Bad break, not included in average.

† Roll edge plates; all the other plates used in the tests had heavy rims.

When the groups are rated according to energy of impact per 0.001 inch thickness of the bottom of the plates, taken as an index of the volume of the plates, the order in which they occur is materially changed. This point will be discussed after the porosity and density data for the plates have been presented.

Porosity Determinations.

From the bottom of each plate broken in the impact tests, a piece weighing about 40 grams was taken for the porosity determinations, dried, and weighed. Saturation with distilled water was conducted by immersing the specimens in boiling water for 5 hours. After this, the weights of the saturated pieces

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in air and suspended in water were determined. The absorption and porosity of each piece were calculated by the usual formulas.[1]

For calculating the bulk specific gravity the following formula was used:

$$\text{Bulk specific gravity} = D/W - S$$

in which

W = the saturated weight in air.
D = the dry weight.
S = the weight of the saturated piece suspended in water.

The term bulk specific gravity is used to avoid confusion, for while the expression "apparent specific gravity" is sometimes used to designate the specific gravity determined by the above formula, it is also applied to specific gravities calculated by a similar formula in which W — S in the above formula is replaced by D — S. The formula used for bulk specific gravity gives the weight of unit volume of a substance as it appears to the eye. The other formula gives the weight of unit volume of what has been called the skeleton of a substance, that is, the material not penetrable by water. We believe Potts and Knollman[2] were the first to use the expression bulk specific gravity.

When a substance is porous, bulk specific gravity can be determined by dividing the percentage porosity by the percentage absorption. That this relationship holds is shown by the equation

$$\frac{W - D/W - S}{W - D/D} = D/W - S.$$

For porous substances, then, the bulk specific gravity shows the ratio of percentage porosity to percentage absorption. Of course, this relationship does not hold true for averaged results.

The data for porosity and allied determinations are shown in Tables 4 and 5. In these tables we see that there was considerable variation in porosity and other values in each lot of semi-porcelain plates, and also in the average values of the various

[1] R. C. Purdy, *Bull.* 9, Illinois Geol. Sur.

[2] *Trans. Am. Ceram. Soc.*, 15, 353 (1913).

lots. In the hotel china plates the variations are, of course, not as great. It is interesting to note that there is seemingly no simple relationship between the data given here and the resistance to impact of the various lots of plates. This is due in large part to the fact that the plates varied in weight, bulk, and design.

TABLE 4.—ABSORPTION POROSITY AND BULK SPECIFIC GRAVITY OF SEVEN-INCH HEAVY SEMI-PORCELAIN HOTEL PLATES.

Factory.	Plate.	Absorptions in percents of dry weights.	Open pores in percents of total volumes.	Bulk specific gravity.
A.....	1	7.75	16.12	2.08
	2	6.44	13.71	2.13
	3	7.80	16.20	2.08
	4	8.18	17.00	2.08
	5	8.05	16.55	2.06
	6	9.42	19.02	2.02
	Average.....	7.94	16.43	2.075
B.....	7	8.09	16.80	2.08
	8	6.27	13.41	2.14
	9	7.87	16.25	2.06
	10	5.47	11.82	2.16
	11	5.03	10.98	2.19
	12	6.06	12.84	2.12
	Average.....	6.465	13.68	2.125
C.....	13	8.99	18.30	2.04
	14	9.24	18.71	2.03
	15	7.54	15.85	2.11
	16	9.39	19.15	2.04
	17	9.50	19.19	2.02
	18	8.56	17.40	2.04
	Average.....	8.87	18.10	2.047
D.....	19	12.05	23.52	1.95
	20	10.70	21.41	2.00
	21	11.73	23.02	1.96
	22	12.26	23.93	1.94
	23	11.82	23.19	1.96
	24	11.24	21.98	1.97
	Average.....	11.63	22.84	1.963

TABLE 5.—ABSORPTION, POROSITY AND BULK SPECIFIC GRAVITY OF SEVEN-INCH HEAVY CHINA HOTEL PLATES.

Factory.	Sample.	Absorptions in percents of dry weights.	Open pores in percents of total volume.	Bulk specific gravity.
E†	25	1.91	4.30	2.26
	26	0.43	1.05	2.38
	27	0.10	0.24	2.37
	28	0.23	0.55	2.34
	29	0.60	1.41	2.37
	30	0.47	1.11	2.36
	Average	0.62	1.44	2.35
F†	31	0.93	2.21	2.39
	32	0.79	1.91	2.40
	33	0.74	1.78	2.40
	34	0.94	2.24	2.38
	35	0.86	2.06	2.40
	36	0.90	2.12	2.36
	Average	0.86	2.05	2.39
G	37	0.0	0.0	2.35
	38	0.06*	0.14*	2.38
	39	0.0	0.0	2.38
	40	0.0	0.0	2.33
	41	0.03*	0.07*	2.35
	41	0.03*	0.07*	2.40
	Average	0.02*	0.05*	2.365

† Roll edge plates; all the other plates used in tests had heavy rims.
* Negligible.

TABLE 6.—RATING OF THE GROUPS OF PLATES[1] ACCORDING TO VARIOUS AVERAGE VALUES.

Rank.	Energy of impact in ft. lbs. of blow producing fracture. Factory.	Value.	Energy of impact in ft. lbs. per cubic inch of plate. Factory.	Value.	Energy of impact in ft. lbs. per pound of material. Factory.	Value.	Open pores in percents of total volume. Factory.	Value.
1	C	1.86	C	0.925	C	1.255	G	0.05
2	G	1.48	G	0.794	G	0.934	E	1.44
3	B	1.37	F	0.721	B	0.926	F	2.05
4	F	1.11	B	0.710	F	0.840	B	13.68
5	D	1.03	E	0.638	E	0.755	A	16.43
6	E	0.97	D	0.493	D	0.696	C	18.10
7	A	0.87	A	0.442	A	0.588	D	22.84

[1] Plates from factories A, B, C, and D were heavy 7-inch semi-porcelain. Those from factories E and F were roll edge 7-inch vitrified hotel china. Those from factory G were 7-inch heavy vitrified hotel china.

From the data in Tables 2–5, it is a simple matter to calculate the energy of impact of the blow producing fracture per unit weight of material in the plates and for unit volume of the plates. These values are of importance for, other things being equal, the trade prefers the lightest and thinnest plates that will give the required service. The calculations have been made for the averages of the plates in each group and are shown in Table 6 together with the rating of the groups of plates according to other average values. The values per cubic inch of plate given in this table are a much more nearly accurate statement of the relation of the volume of the plates to resistance to impact than those derived from the thickness of the bottoms of the plates and shown in Table 2.

SUMMARY.

1. It is possible to devise comparatively simple tests to determine the mechanical properties of such ceramic products as table wares.

2. The resistance to impact of the various makes of plates varied widely.

3. The vitrified hotel china plates tested were not superior as a class to the semi-porcelain plates in resistance to heavy impact blows. However, they did not chip with light blows while the semi-porcelain did.

4. There was no direct relation discernible between porosity and resistance to impact.

5. The design of plates undoubtedly is an important factor in determining their resistance to impact.

AMERICAN CERAMIC SOCIETY.

Acquisition of New Members during March, 1919.

Resident Associate.

Acheson, A. E., J. H. Gautier & Co., Jersey City, N. J.
Acki, S., Libbey-Owens Sheet Glass Co., Charleston, W. Va.
Algeo, A. M., Hazel-Atlas Glass Co., Washington, Pa.
Binns, Norah W., Alfred, N. Y.
Bowman, Geo. H., 1931 E. 93rd St., Cleveland, Ohio.
Bracken, Lloyd, 770 Coleman Ave., Clarksburg, W. Va.
Brooks, Charles T., 8205 Euclid Ave., Cleveland, Ohio.
Cake, B. F., Denny-Renton Clay & Coal Co., Renton, Wash.
Coe, John H., 547 Rosedale St., Wilkinsburg, Pa.
Cruikshank, J. W., 1206 Hartje Bldg., Pittsburgh, Pa.
Cunning, W. E., West End Pottery Co., East Liverpool, Ohio.
Harvey, J. Ellis, Orviston, Pa.
Hewitt, L. C., Laclede-Christy Co., St. Louis, Mo.
Hill, Jas. H., 706 Equitable Bldg., Los Angeles, Cal.
Houser, A. T., Wood-Lloyd Co., Pittsburgh, Pa.
Lemley, W. E., Denny-Renton Clay & Coal Co., Taylor, Wash.
Lloyd, A., Hazel-Atlas Glass Co., Washington, Pa.
Martin, Leonard A., Denny-Renton Clay & Coal Co., Seattle, Wash.
Pickett, Thos. F., Hazel-Atlas Glass Co., Washington, Pa.
Reddick, W. L., Hazel-Atlas Glass Co., Grafton, W. Va.
Richardson, E. A., Glass Technology Dept., National Lamp Wks., Cleveland, Ohio.
Saunders, Arthur E., Arkansas Glass Products Co., Van Buren, Ark.
Strong, Thos. L., Strong Mfg. Co., Sebring, Ohio.
Tait, Howard J., Phoenix Glass Co., Monaca, Pa.
Troutman, Frank E., Standard Plate Glass Co., Butler, Pa.
Wells, R. D., Floyd Wells Co., Royersford, Pa.

Foreign Associate.

Carter, Charles, Carter Encaustic Tile Wks., Poole, England.
Eilers, H. F., Dominion Glass Co., Wallaceburg, Canada.
Gibson, M. F., National Fireproofing Co., Toronto, Can.
Searle, Alfred B., The White Building, Sheffield, England.
Sieurin, Emil, Hoganas, Sweden.

Corporation.

Jones Hollow Ware Co., Baltimore, Md.
Carborundum Company, Niagara Falls, N. Y.

AMERICAN CERAMIC SOCIETY.

Associates Elevated to Active Membership at the Annual Meeting in Pittsburgh, February 5, 1919.

Resident.

Acheson, E. G.
Ashbaugh, Chas. C.
Bacon, Raymond F.
Bainbridge, W. L.
Barton, G. E.
Bell, M. L.
Bentley, L. L.
Berry, C. W.
Bole, G. A.
Booze, M. C.
Bowman, W. J. J.
Bryan, M. L.
Campbell, A. R.
Carder, F. R.
Cermak, Frank.
Child, J. L.
Clark, A. B.
Clark, Wm. M.
Conkling, S. O.
Cook, Chas. H.
Danielson, R. R.
Davis, John B.
Day, Arthur L.
Dornbach, Wm. E.
Duval, A. L.
Ellerbeck, Wm. L.
Fackt, Geo. P.
Frost, Leon, J.
Fulper, Wm. H.
Gillinder, James.
Gorton, A. F.
Greener, Geo. C.
Gregory, M. E.
Grueby, Wm. H.
Haaf, Geo.
Hall, Robert T.
Hansen, Abel.
Henry, Frank R.
Hipp, Wm. G.
Hoffman, Geo. E.
Hood, B. Mifflin.
Hornung, Martin R.
Jacobs, Wm. M.
Jeffery, Joseph A.
Jensen, Chas. H.
Jeppson, Geo. N.
Kalbfleisch, G. C.
Kanengeiser, Fred R.
Kerr, W. B.
Knight, M. A.
Kohler, W. J.
Landers, W. F.
Linbarger, S. C.
Maddock, A. M.
Maddock, John.
Malsch, Werner.
Maynard, T. Poole.
Mellor, F. G.
Metzner, Otto.
Middleton, Jefferson.
Mossman, P. B.
Muckenhirn, Chas. H.
McDougal, Taine G.
McDowell, J. S.
McElroy, R. H.
Ortman, Fred B.
Owens, Francis T.
Peregrine, C. R.
Poole, Joshua.
Rankin, G. A.
Rea, Wm. J.
Rhead, Frederick H.
Richardson, Ernest.
Riess, D. F.
Rusoff, Samuel.
Ryan, J. J.
Salisbury, B. E.
Sanders, John W.
Sant, Thomas H.
Scholes, Samuel R.
Seaver, Kenneth.
Sebring, Chas. L.
Shoemaker, Geo. W.
Singer, L. P.
Smith, James M.
Solon, Marc.
*Sosman, Robert B.
Staudt, August.
Steinhoff, F. L.
Sullivan, E. C.
Sweely, B. T.
Taylor, Royal W.
Taylor, Wm. C.
Tone, Frank J.
Townsend, Everett.
Treischel, Chester.
Truman, Gail R.
Walden, A. S.
Will, Otto W.
Wilson, Hewitt.
Zimmerli, Wm. F.

Foreign.

Carter, Owen.
Cobb, John W.
Cox, Paul E.
Luze, Henry de.
Emory, George.
Fredriksson, Nils.
Haviland, Jean.
†Lundgren, H. J.
Lusby, C. A.
Mellor, J. W.
Sailly, Paul.
Turner, W. E. S.
Walker, E. E.

* Elevated, March, 1919. † Died February, 1919.

ACTIVITIES OF THE SOCIETY.

The following motions have recently been passed by the Board of Trustees:

March 3rd. It was voted to elevate Dr. Robert B. Sosman, of the Geophysical Laboratory, Washington, D. C., to Active Membership.

March 10th. It was voted to extend indefinitely the authorization of the Secretary to accept applications for Associate and Corporation memberships.

March 20th. It was voted to grant the Membership Committee the sum of $200 for its use in soliciting new members.

March 20th. It was voted to appropriate a sum not to exceed $2,000 for the conduct of new departments in the JOURNAL.

March 25th. It was voted to publish the minutes for 1918 at once instead of in the Year Book.

March 28th. It was voted to retain the membership of the Society in the National Fire Protection Association, with the suggestion that a committee be appointed by the President to represent the Society in this Association.

AMENDMENTS TO THE RULES.

The following amendments to the Rules have been adopted by letter ballot:

SECTION II—"Membership"—Shall be amended to read:

(1) The Society shall consist of Honorary Members, Active Members, Associate Members, Affiliated Members, and Corporation Members.

(2) Corporation Members must be persons, firms, or corporations who, being interested in the Society, make such financial contributions for its support as are prescribed in Section III.

(3) The Board of Trustees shall in each regular meeting advance to Active Membership all Associate Members whose activity in the Society or whose prominence in the ceramic industries, in the opinion of a majority of the Board of Trustees, merits such recognition. Any Active Member may nominate in writing Associates for Active Membership and the Board of Trustees shall act on such recommendation at the next regular meeting of the Society. A written statement of the qualifications of each candidate shall be presented at that meeting of the Society and published promptly in the JOURNAL.

(4) Corporation members may be enrolled on the appropriate list of the Society at any time upon payment of the dues prescribed in Section III.

(5) All Honorary Members, Active Members, Associate Members, and Corporation Members shall be equally entitled to the privileges of member-

ship, except that only Active Members and one representative of each Corporation member shall be entitled to vote. Such representative shall be officially designated by the person, firm or corporation represented. Only active members shall be entitled to hold office. The roster of each grade of membership shall be printed separately in at least one publication issued by the Society annually.

(6) Any person may be expelled from any grade of membership of the Society if charges signed by five or more active members be filed against him or her, and if the Board of Trustees examine into and sustain said charges by a majority vote. Such person, however, shall first be notified of the charges against him and be given a reasonable time to appear before the Board of Trustees or to present a written defense, before final action is taken.

SECTION III—"Dues"—Shall be amended to read:

(7) On and after February 1, 1920, no initiation fee shall be levied or collected from Associates upon promotion to Active Membership. The annual dues for Active Members shall be fixed by the Board of Trustees, but shall not exceed ten dollars.

(8) Corporation Members shall pay no initiation fee. The annual dues shall be fixed by the Board of Trustees but shall not be less than twenty-five dollars. The privileges of membership shall begin upon payment of the annual dues.

SECTION IV—"Officers"—Shall be amended to read:

(9) The affairs of the Society shall be managed by a Board of Trustees consisting of the President, the Vice-President (an Honorary Secretary, if there be one), the Treasurer, the retiring president and his immediate predecessor, and three trustees. The President, the Vice-President and the Treasurer shall be elected to serve one year. The Trustees shall be elected to serve three years, one Trustee being elected each year.

(10) The President shall have general supervision of the affairs of the Society under the direction of the Board of Trustees and shall perform such other duties as pertain to his office.

(11) He shall countersign the checks drawn by the Treasurer when such drafts are known by him to be proper and duly authorized by the Board of Trustees.

(12) A Secretary shall be appointed annually by the Board of Trustees to serve for one year.

(13) He shall see that all moneys due the Society are carefully collected and deposited in a Bank approved by the Board of Trustees, and he shall transfer monthly such funds to the custody of the Treasurer. The Secretary shall carefully scrutinize all expenditures and use his best endeavors to secure economy in the administration of the Society. He shall personally investigate and certify the accuracy of all bills or vouchers on which money is to be paid. He shall have charge of the books of account of the Society and shall furnish monthly to the Board of Trustees a statement of monthly balances. He shall present annually to the Board of Trustees a balance sheet of his books as of

the 31st of December and shall furnish from time to time such other statements as may be required of him.

(14) The Secretary shall furnish a suitable bond for the satisfactory performance of his duties, which shall be held in the custody of the President.

(15) The Treasurer shall receive all moneys due the Society, and deposit the same in the name of the Society in a National Bank designated by the Treasurer and approved by the Board of Trustees. He shall invest all funds not needed for current disbursements, as shall be ordered by the Board of Trustees. He shall pay all bills by draft, when certified by the Secretary or as ordered by the Board of Trustees. He shall furnish a satisfactory bond for the proper performance of his duties, which shall be held in the custody of the President.

(16) All securities belonging to the Society shall be held in the custody of the Treasurer.

(17) The accounts of the Secretary and of the Treasurer shall be audited before each annual meeting by a certified public accountant or some other competent person designated by the Board of Trustees.

SECTION V—"Elections"—Shall be amended as follows:

(18) At the annual meeting a nominating committee of five active members, not officers, of the Society, shall be appointed.

(19) At least ninety days before the annual meeting this committee shall send the names of the nominees to the Secretary who will immediately send a copy of the same to each Active Member. Any five Active Members may act as a self-constituted Nominating Committee and present the names of any nominees to the Secretary, provided this is done at least thirty days before the annual meeting. The names of all nominees, provided their assent has been obtained before nomination, shall be placed on the ballot without distinction as to nomination by the regular or self-constituted Nominating Committee and shall be mailed to each Active Member, not in arrears, at least twenty days before the annual meeting. The voting shall be confined to the names appearing on this ballot. The ballot shall be enclosed in an envelope on which there shall be no mark for identification other than the word "Ballot." This envelope shall be enclosed in another envelope for mailing, addressed to the Secretary, upon the back of which the voter shall endorse his name. The ballot envelope shall be opened in the presenee of three scrutineers appointed by the President, who will report the result of the election at the last session of the annual meeting. A plurality of affirmative votes cast shall elect.

SECTION VII—"Standing Committees"—Shall be amended as follows:

Standing Committees

(20) 5, "Local and Student Sections" to be
"Sections and Divisions"

(21) 8, "Military and Economic Preparedness" to be
"Research and Development"

(22) The Committee on Sections and Divisions shall consist of five mem-

bers. Its duties shall be to promote the organization of Local Sections, Student Branches, and Divisions.

(23) The Committee on Research and Development shall consist of five members. Its duties shall be to organize and encourage scientific investigations pertaining to the silicate and related industries, especially those investigations which will stimulate the development of our national industries and resources. It shall be empowered to coöperate with similar committees of other scientific societies and with Government bureaus.

SECTION VIII—"Divisions"—Shall be amended as follows:

(24) The affairs of a Division shall be managed by a Chairman, a Secretary-Treasurer, and a Council of four members. The officers of the Division and two members of the Council shall be elected annually by ballot at the last session of the Division held during the annual meeting of the Society and shall take office at the close of the meeting at which they are elected. All members of a Division are entitled to vote. The members of the Council shall hold office for two years. The officers shall hold office for one year or until their successors are elected. The Board of Trustees shall fill any vacancies occurring through death or resignation among officers of a Division.

(25) The Secretary-Treasurer of each Division shall make an annual report to the Society of the activities and finances of the Division.

SECTION XI—"Publications"—Shall be amended as follows:

(26) One copy of each issue of the paper-bound edition of the JOURNAL shall be sent prepaid to each member of the Society not in arrears. No member shall be furnished with more than one copy of each issue free for any single year. A member may be permitted to complete by purchase one file of the publications of the Society at less than the current commercial rate, the amount to be fixed by the Board of Trustees and to be called the member's rate.

NOTICE TO MEMBERS.

The January, February and March numbers of the JOURNAL will be sent to members whose dues for the past year have been paid. If the dues for the current year are then unpaid the subscription will be discontinued.

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EDITORIALS.

THE PATENT OFFICE AND THE PATENT SITUATION.

By George E. Middleton, an Assistant Examiner, U. S. Patent Office.

In view of the editorial on "The Patent Situation" in the September number of the Journal of the American Ceramic Society,[1] in which our patent system, and particularly the Patent Office, was so bitterly arraigned, it has seemed advisable to submit the following, partly by way of defense of those charged with the administration of the Patent Office, but more particularly by way of a general survey of the field, pointing out some of the reasons why the office has, in a measure, failed to fulfill its high duty, and some of the reforms now in contemplation. This is done with the hope that the American Ceramic Society may, the more effectively, lend its influence in the achievement of these reforms.

The industrial development of America under the present patent system administered through the Patent Office, has certainly justified the judgment of its founders. But the industrial arts have progressed faster than the system designed to promote that progress. Such a situation cannot long endure and it is becoming increasingly evident to those conversant with the facts that reform is urgent if the system is to continue to justify its existence. The problem is a big one, but not impossible of solution.

Historical.

The framers of the Constitution appreciated the wisdom of encouraging invention by the grant of some suitable reward to

[1] *J. Am. Ceram. Soc.*, 1, 59 (1918).

inventors. It was first proposed that: "The Congress shall have power to encourage by premiums and provisions the advancement of useful knowledge and discoveries"—but this was abandoned in favor of a monopolistic grant, the provision adopted being—"The Congress shall have power * * * * to promote the progress of science and the useful arts by securing for limited times to * * * inventors the exclusive right to their * * * discoveries." (Art. I, Sec. 8.) There are two systems under which this monopoly may be secured. Under one, the applicant is granted his patent as a matter of course, the Patent Office acting merely as a recording or registration bureau, the question of validity being left entirely with the courts. This is known as a registration system. The other requires an examination into the merits of the alleged invention by a corps of experts and the grant of the patent carries with it *prima facie* presumption of validity. This is known as an examination system. Under a registration system, the burden of proving validity is upon the patentee and his is the responsibility for claiming no more than he is justly entitled to. If he claims too much, his whole patent may be declared invalid even though some of his claims may be limited to his real invention. Under an examination system, the burden of proving invalidity is upon the infringer and as the Patent Office itself has essayed to pass upon the claims, patents are never declared wholly void because some of the claims may be too broad.

Both systems have been tried in this country. Acting under its constitutional authority, Congress passed the first patent act in 1790. The system thus established was an examination system and so rigorous was the examination and so high the standards set for those entitled to the grant, that during the three years of its existence only 60 patents were issued. The industries complained loudly against this severity and in 1793 the original act was superseded by another. The pendulum had swung to the other extreme for the system under the act of 1793 was wholly a registration system. This obtained until 1836, but proved grossly unfitted to the needs of the country and was fruitful only of abundant ligitation, fraud and confusion. By 1835, conditions had grown so intolerable that John Ruggles, a senator from

Maine, moved that the Senate appoint a committee to investigate the patent situation. This was done and Ruggles was made Chairman of the Committee which made its report in April, 1836, and at the same time submitted a plan for the reorganization of the Patent Office. The indictment of the registration system returned by Ruggles in this report contains some significant charges—significant because substantially the same as those heard today directed against our present system. He said in part:

"The extent of the evils resulting from the unrestrained and promiscuous grants of patent privileges may be imagined when it is considered that they are now issued, since this year commenced, at the rate of more than a thousand a year; a considerable portion of which are doubtless void for want of originality in the inventions patented, either in whole or in some of the parts claimed as new.

"A necessary consequence is that patents, even for new and meritorious inventions, are so much depreciated in general estimation that they are of but little value to the patentee, and the object of the patent laws, that of promoting the arts by encouragement, is in a great measure defeated.

"To prevent these evils in future is the first and most desirable object of a revision and alteration of the existing laws on this subject. The most obvious, if not the only means of effecting it, appears to be to establish a check upon granting of patents, allowing them to issue only for such inventions as are in fact new and entitled by the merit of originality and utility to be protected by law. * * * * "

This arraignment evidently had its effect, for before the end of the year, Ruggle's plan had been placed upon the statute books and remains today, except for minor changes, our fundamental law of patents and the foundation of our present examination system.

Thus we see that the registration system was tried in this country for more than forty years, that it was found wanting and was superseded by an examination system under which patents do not issue as a matter of course, but only after an examination which convinces those in authority that the applicant has truly invented something new and useful. That an effective examination system is superior to one based on registration is pretty generally conceded and it is noteworthy in this connection that among the great industrial nations of the earth today, only one—France—still adheres to the old registration scheme.

The Functions of the Patent Office under Our Examination System.

The functions of a patent office under a registration system are relatively simple as the duty of passing upon the merits of an invention is thrown upon the courts. It is obvious that under an examination system, a patent office must be a much more complex institution. The Revised Statutes provide that:

"Any person who has invented or discovered any new and useful art, machine, manufacture, or composition of matter, or any new and useful improvements thereof, not known or used by others in this country, before his invention or discovery thereof, and not patented or described in any printed publication in this or any foreign country, before his invention or discovery thereof, or more than two years prior to his application, and not in public use or on sale in this country for more than two years prior to his application, unless the same is proved to have been abandoned, may, upon payment of the fees required by law, and other due proceeding had, obtain a patent therefor (Sec. 4886).

"Before any inventor or discoverer shall receive a patent for his invention or discovery, he shall make application therefor, in writing, to the Commissioner of Patents, and shall file in the Patent Office a written description of the same, and of the manner and process of making, constructing, compounding, and using it, in such full, clear, concise, and exact terms as to enable any person skilled in the art or science to which it appertains, or with which it is most nearly connected, to make, construct, compound, and use the same; * * * * and he shall particularly point out and distinctly claim the part, improvement, or combination which he claims as his invention or discovery." (Sec. 4888.)

There are then, so far as the merits of the case go, four essential prerequisites to the grant of a patent. The applicant must have:

1. *Invented* something.
2. It must be *new*.
3. It must be *useful*.
4. It must be *fully and clearly* disclosed in the application and *distinctly claimed*.

It is further provided:

"On the filing of any such application and the payment of the fees required by law, the Commissioner of Patents shall cause an *examination* to be made of the alleged new invention or discovery; and if on such examination it shall appear that the claimant is justly entitled to a patent under the law, and that the same is sufficiently useful and important, the Commissioner shall issue a patent therefor." (Sec. 4893.)

This examination must, therefore, concern itself in the main with the four essential prerequisites enumerated above, and it is the simple function of the Patent Office to conduct such examination and to grant or refuse to grant patents in the light of its results. But an honest study of an application from these four points of view is neither a simple nor an easy matter.

Invention.—Granting novelty and utility and all the rest, has the applicant exercised that indefinable something known as the inventive faculty? If he has done no more than the skilled mechanic might well have done under the circumstances, if he is a mere technical Philistine, then the grant should be withheld. The patent laws wisely extend protection only to *inventors*. The determination of this question requires a clear judgment, together with an understanding of the development of the particular art. Of course standards vary, not only from time to time, but among individual examiners. From 1790 to 1793 the standard was probably too high; today it is probably too low and must continue so as long as the constant pressure of incoming applications makes it imperative that they be got rid of with greater dispatch than is consistent with careful thought.

Novelty.—This is a purely objective test. In the last analysis, determination of invention is a matter of opinion, but not so novelty. The claim to novelty admits of concrete disproof and it is the duty of the examiner to search for the evidence. If the alleged invention was described in a printed publication anywhere, in any tongue, at any time prior to the filing date of the application, the examiner, if functioning properly, should find it and reject the claims upon it. If the alleged invention was publicly known or used, or on sale by others in this country before the filing date of the application, even though never described in a printed publication, the examiner, if he be a thoroughly efficient examiner, should know of it and reject the claims accordingly. Certainly the main work of the Patent Office today is the determination of this question of novelty. It is this which involves "the search." No expert, however wise short of omniscience, can determine novelty without a search of the prior art, and the value of his determination will be in direct proportion to the thoroughness of that search.

Utility.—Greater than invention, greater than novelty, utility is hardly considered by the Patent Office. The Statutes direct the Commissioner to issue a patent when, upon examination, it shall appear that the alleged invention is *sufficiently useful and important.* And why not? The whole patent scheme was created to promote the progress of the *useful* arts. But how can the academic Patent Office pass upon utility? The truth is that it cannot and does not. Where the alleged invention is frivolous, or obviously inoperative or injurious to public health or morals, a patent is refused, but in other cases, the test of utility is left for the tribunal of experience. On this issue, the applicant, like the pleader at common law, is willing to put himself upon the country. Rejections on the ground of inoperativeness are not infrequent, but the judgment of the examiner is based on purely *a priori* considerations where all the conditions set forth by the applicant are not old. For years, claims drawn to heavier-than-air flying machines were complacently rejected on the ground that such machines were impossible! The office, absolutely without means of making the simplest tests for itself and realizing the occasional folly of academic reasoning, has, on the whole, however, fallen into the unfortunate habit of resolving doubts on the score of inoperativeness in favor of the applicant. The present method of considering utility is not much different from that employed under a registration system.

Disclosure and Claims.—A patent is a contract, the inventor and the public being the high contracting parties. The inventor discloses his new and useful invention to the public, teaching it its principles and its application; the public, in consideration for this disclosure, grants to the patentee for the term of seventeen years, the exclusive right to make, use and vend the subject-matter of the invention If the subject-matter of the patent is neither indicative of invention, nor new, nor useful, it is clear that the patentee has failed to give consideration for his monopoly and that the contract is void. But this fourth element of the patentee's consideration, disclosure, is of equal importance. If the public is not clearly and accurately taught how to avail itself of the invention, of what value is it? It is, therefore, the duty of the examiner to see that no essential information is omitted,

that each patent is so full and complete in itself that anyone skilled in the art to which it pertains can readily apply the invention, guided by the specification. Where doubt arises as to the completeness of disclosure, tests should be made, but these the Patent Office is without facilities to make.

In describing his invention, an inventor must, of necessity, give it a setting in the prior art. Therefore, the relation of the invention to the prior art must be set forth. But this description is not sufficient to define the invention and, from the specification alone, it would be impossible for the public to know exactly the scope of the monopoly which is granted. For this reason the description or specification must conclude with a specific claim or claims defining the metes and bounds of the invention. It is the duty of the examiner to see that these claims embrace neither more nor less than the applicant is entitled to. Under a registration system, this is not so, the applicant himself assuming the burden of claiming no more than his invention.

A true examination system then differs from one based solely on registration mainly in the method of passing upon invention, novelty and utility, and where, from one cause or another, consideration of invention becomes perfunctory, determination of novelty superficial, and judgment upon utility left for the High Court of Experience, the system is substantially a registration system. This is the crux of the whole matter.

Some Reasons Why the Patent Office Has Failed to Function Successfully.

The failure of the office to cope successfully with the problem is not strange. The industrial development of the world within the past fifty years is among the striking phenomena of history and in this development America has taken the lead. The Patent Office is the pulse of this quickened industrial life. To date, 1,300,000 patents have been granted and by the close of the decade another 100,000 will easily have been added. The million mark was passed only in 1911. This means that in this present decade 400,000 of the 1,400,000 patents will have been granted, or 28 per cent of the patents in 12 per cent of the time, and the end is not yet. It required 75 years to complete the first million,

the second will undoubtedly be rounded out in 20, and so on, *ad infinitum*.

The number of patents granted by foreign countries has also increased similarly until today the total approximates 2,700,000. At the same time, the mass of scientific and technical publications has grown to a prodigious size and is constantly increasing with accelerating speed. And then besides these printed evidences of scientific and industrial growth, there is another and perhaps a vaster fund of knowledge and experience resident in unwritten and unpublished shop and laboratory practice—things so well known to the workers that no one has deemed it worth while to mention them in print, and yet these are the very fundamental things that keep the wheels of earth turning. Indeed, the sum total of human knowledge in this one field of industrial arts is something stupendous—certainly enough to discourage the most erudite examiner of patents.

The examining force of the office has been slightly increased to keep pace with this mighty advance, but the increase has been ridiculously inadequate. In 1890, the corps comprised 159 assistant examiners (the ones who make the searches) and in 1917, 360. In 1890, the number of applications received was 41,048, and in 1917, 70,373. This means 258 new applications examined per man in 1890 and only 195 in 1917, *but*, the field of search in 1890 was much smaller than in 1917. In the former year, the number of United States patents then granted was 443,000 and the available foreign patents 635,000, making a total of 1,078,000. At the close of 1917, there were 1,251,000 United States and 1,770,000 available foreign patents, making the available field of search in patents alone 3,021,000. The work done by an examiner in novelty searches varies directly as the product of the number of applications received multiplied by the field of search, and inversely as the number of assistant examiners. The work done by an examiner in his consideration of an application aside from the search varies directly as the number of applications and inversely as the number of assistant examiners. An examiner's time is about equally divided between searching and these other duties. These two ratios may be designated as the index of work of their respective kinds. In any given year, if A = applications

received, S = the field of search, E = the assistant examiners, W′ = the index of search work, and W″ = the index of work other than searching, then

$$W' = \frac{A \times S}{E} \qquad W'' = \frac{A}{E}.$$

Expressed in tabular form, reducing the indices of work for 1890 to 100 and taking the mean of these as W, the true index of work, the above figures reduce to

	A.	S.	E.	W′.	W″.	W.
1890	41,048	1,078,000	159	100	100	100
1917	70,373	3,021,000	360	279	72	175

In other words, as the time spent in examinations does not vary materially, the quality of the work done in 1917 was about four-sevenths of that in 1890. In order to have maintained the quality of 1890, it is obvious that the number of assistant examiners in 1917 should have been 75 per cent greater than it was, or 630.

It is to be observed that these figures utterly fail to take into account either the scientific and technical literature or the known shop practice, both of which, as above pointed out, have increased enormously since 1890. The consideration of these elements would vastly increase the field of search factor for 1917 as compared with that of 1890 and so correspondingly increase the index of work for 1917. It is also to be borne in mind that the taking of the figures for 1890 as a basis for comparison is by no means an assumption that conditions at that time were ideal. They were undoubtedly far from it. The comparison merely shows that conditions are growing worse.

The total average time given by an examiner to each application based on the 1917 figures is about nine and one-half hours. Not more than half of that time is given to actual searching; the other half is consumed in reading the case, noting informalities, writing the necessary letters, studying new claims presented by amendment, etc. And as this nine and one-half hours represents the quotient of the examiner's *entire* time divided by number of applications, any stray moments devoted to an attempt to keep in touch with the development of his art must be deducted from

it. Validity searches require weeks. Is it any wonder that validity searchers sometimes discover references overlooked by or unknown to the examiner? As a conservative estimate based on the foregoing data, the present examining corps should be doubled.

The Patent Committee of the National Research Council in their report to be mentioned later, state:

"The patents granted by the United States Patent Office are of less average probable validity than formerly because the number of applications for patents and the field of search are constantly increasing, while the examining force for many years has been insufficiently large and has not been increased proportionately. The inducements are so unattractive that 25 per cent of the examining force has resigned within the past three years. * * * *

"The work of the Patent Office has grown so much more rapidly than has the examining force that the examination to determine whether or not the invention claimed in an application for patent is novel is imperatively restricted to the field of search where it is most likely that the invention will be found. *Many patents are granted which would not be granted if the examiner had time to make a thorough search.* * * *"

It is not possible to estimate the probable validity of the patents now granted. In the adjudicated patents reported in the Official Gazette of the Patent Office for 1918, the issue of validity was raised in suits involving 135 patents and of these, 75 were declared valid, and 60, or 44 per cent, invalid, either in whole or as to some of the claims. Of these 60, at least 55 were invalidated for lack of invention in view of the prior art. This percentage of invalid patents for 1918 seems high, but it must be borne in mind that during that year there were 587,000 unexpired patents in existence. Many of these were so limited, unpractical, or out of date, that they would never get into litigation and many so clearly recognized as valid that they also would never appear in court. No one can say what the average probable validity of the patents now granted is, but in view of the necessarily limited search and the tendency to lower standards of invention now prevailing, it must be, as the National Research Council pointed out, less than formerly. The noteworthy thing, however, is not that invalid patents are granted, but that examiners circumscribed and limited in the exercise of their duties as the present examiners certainly are, should be able to issue so many patents of proved validity.

Some Proposed Reforms.

The writer of the editorial in question believes that the situation, bad as it is, admits of easy solution. He proposes a remedy which is "neither difficult nor complex" and which consists in the simple "creation of a small committee of experts for each of the natural divisions of industry and technology, among them one for the field of ceramics." If applications were filed, a score a week, this small committee of experts might develop into a highly efficient and satisfactory examining corps, but how could they cope with a flood of 1500 applications a week? The present examining corps—a committee of 400 experts—finds itself unequal to the task. As previously pointed out, a search of the prior art is necessary before the simplest idea can be patented. No committee of experts can possibly pass upon novelty out of the fullness of their experience alone and is it to be expected that they could search with much greater efficiency and speed than the present corps? It is feared that the proponent of this apparently simple remedy has failed to grasp the magnitude of the task with which the Patent Office finds itself confronted.

In 1917 the Patent Office Society was organized with the object, as set forth in its constitution, "to further the industrial development of the United States in so far as the patent system is a factor thereof, to promote and foster a true appreciation of the American patent system, to cultivate a high standard of professional ethics among patent practitioners and examiners, and to promote the professional, intellectual and social welfare of the members of the Society."

The first fruits of the Society's activities have developed through its affiliation with the National Research Council. This Council in conformity with a request embodied in a resolution adopted by the Patent Office Society and fully concurred in by the Commissioner of Patents, appointed a committee for the study of Patent Office problems. That committee consisted of five scientists and three members of the Patent Bar Association, all men of national prominence in their chosen fields. They were Dr. William Durand, Dr. Leo H. Baekeland, Dr. M. I. Pupin, Dr. R. A. Millikan, Dr. S. W. Stratton, Dr. Reid Hunt, Mr. Frederic

P. Fish, Mr. Thomas Ewing and Mr. Edwin J. Prindle. Mr. Ewing was Commissioner of Patents from 1913 to 1917. The recommendations of this committee may be epitomized as follows:

1. A Single Court of Patent Appeals.
2. The Patent Office a separate institution, independent of the Interior Department.
3. Increase in force and salaries of the Patent Office.
4. Compensation for infringement of patents.

A bill embodying the statutory changes necessary to put these recommendations into effect has been drafted and will be presented to Congress in the near future.

Recommendations 1 and 4, although of great importance, do not directly affect the Patent Office and the granting of patents and so will not be discussed in this paper. Recommendations 2 and 3 do affect the Patent Office.

The establishment of the Patent Office as a separate institution would be the first step toward that larger usefulness which the office believes to be its rightful destiny. It was only a few years ago that the Department of Agriculture was itself a minor section of the Patent Office. Witness its position today—a great government department comprising many specialized bureaus. And yet the Department of Agriculture is charged only with the duty of promoting the progress of one industry. The Patent Office, charged with the duty of promoting the progress of *all* science and *all* the industrial arts, is but a small bureau of the Interior Department. The situation is anomalous.

The need for an increase in the force of the Patent Office is obvious, as formerly pointed out, if only to maintain past standards. If improvement is sought, the need becomes imperative. But an increase in the force alone is not a cure-all. The equipment is insufficient. The office has no laboratory of any kind nor is there any machinery by means of which it can adequately coöperate with other government bureaus possessing this equipment. The library facilities are insufficient. The scientific library of the Patent Office ought to be second to none and yet Congress allows only three thousand dollars a year for the Patent Office to keep up with all the technical literature of all the arts. The examiners are too academic and a mere increase

of the corps will not permanently better this. It should be a duty of the examiners to acquaint themselves with the actual practice in the arts which they are examining by periodic visits to the manufacturing establishments of the country. But such visits are very rare. There is an annual fund of five hundred dollars drawn on for this purpose—about $1.25 (one dollar and a quarter) per examiner! But the addition of 200 assistant examiners, as proposed by the National Research Council, would be a blessed relief. It would enable each examiner to spend fifty per cent more time on each application and so tend to make his work just fifty per cent more thorough and efficient than it can be today.

Reforms cannot be effected in a minute. Although some of us feel that the recommendations of the Patent Committee do not strike deeply enough, they certainly represent a long step forward and with a few minor suggestions, have received the approval of the Patent Office Society. We take this opportunity of commending them to the American Ceramic Society for consideration. If "our whole system of affording patent protection to inventions . . . is without rhyme or reason;" if, "like heaven's mercy, patents fall both upon the just and the unjust and the result is injustice and chaos," the fault does not lie with the Patent Office, but with those who refuse to grant it the equipment and the men without which it can not hope to do its great and useful work.

That the present patent system is defective, no one will deny, but on the other hand, it is not quite so bad as the writer of the editorial in question would have us to believe. He asks whether or not patents have been issued upon the employment of kaolin, feldspar, and quartz in ceramic bodies, and then answers the question in the affirmative. Unfortunately, no patent was cited, but a search in the appropriate class and sub-class has failed to reveal any such patent. It is true that patents have been granted on body mixtures, *containing* those rare ingredients, but there were other things present. Whiteware manufacturers using that ancient trinity of the tri-axial diagram need have no fears of infringement suits. The truth is that errors are often made in construing patent claims by those unfamiliar with such matters, and a claim which may at first glance appear very broad and drawn to

some old and unpatentable composition or device, will often, upon closer scrutiny, prove to be in truth limited to certain details which leave the broad field unrestricted. In criticizing the grant of a patent, the critic should first make sure that he appreciates the exact scope of the matter claimed.

There is another phase of the matter. It takes two to make a patent—the inventor and the Patent Office. If "weird compositions of glasses, enamels and glazes" and kilns and driers "representing ideas hoary with antiquity" are patented, it is to be regretted, but it must not be forgotten that it was a ceramist in the first instance who applied for these patents and took oath that the ideas involved were useful and that he believed them to be new. A cursory inspection of the names of recent patentees in the ceramic arts reveals many of eminence in that field.

THE TWENTY-SECOND ANNUAL MEETING.

The Board of Trustees has voted to hold the Twenty-second annual meeting of the American Ceramic Society at Philadelphia, Pa., February 23–26, with headquarters at the Hotel Walton. The Board is to be commended for arriving at an early decision as to the time and place for holding this meeting. The enlarged membership and the increased scope of the activities of the Society have multiplied the preparations necessary for the annual meeting, and with the early start afforded the Philadelphia convention should surpass, if possible, the banner one held in Pittsburgh last February.

The Society has not held an annual meeting in Philadelphia since 1907. With its excellent railroad facilities and hotel accommodations, it would appear that the selection of Philadelphia as a meeting place is a happy one.

ORIGINAL PAPERS AND DISCUSSIONS.

DEVITRIFICATION OF GLASS.

By N. L. Bowen.

All ordinary glasses are undercooled liquids and as such are unstable. Were the stable condition established all such glasses would be completely crystalline at ordinary temperatures. At these temperatures, however, the rate of establishment of the stable condition, that is, the rate at which crystallization proceeds, is absolutely negligible. At higher temperatures, the rate of crystallization (devitrification) may be great enough to become a seriously troublesome factor to the glassmaker. In the literature of glass technology there occur frequent statements that indicate much confusion of ideas concerning the nature of devitrification. Before describing the specific examples of this phenomenon that have been encountered in the course of the writer's experience in the manufacture of optical glass, it seems desirable to discuss the fundamental principles underlying crystallization or devitrification.

Definite chemical compounds almost invariably have definite melting points and the solid, crystalline substance when raised to the melting temperature usually passes promptly into the liquid condition. If the liquid is then allowed to cool and the conditions are such that equilibrium is attained, crystallization will take place at the temperature of the melting point. Melting is therefore a reversible phenomenon and the melting point of a substance is defined as the temperature at which the crystalline and liquid phases are in equilibrium. It is well known, however, that in many chemical and physical changes equilibrium is not always readily established, and in the case of cooling a molten

substance it is frequently possible to lower the temperature below the melting point without the incidence of crystallization, when the liquid is said to be undercooled.

The ease with which undercooling of the liquid can be accomplished varies enormously from one substance to another. Molten metals, for example, usually crystallize promptly when cooled to the melting point. As a rule, water behaves similarly, but with certain precautions, such as the exclusion of dust particles, bubbles, etc., water can be undercooled. Then, again, there are a great number of substances for which no particular precautions are necessary and which normally undercool. Usually, however, only a certain amount of undercooling can be accomplished and when this has been attained crystallization occurs. There are, however, certain substances that can be undercooled an indefinite amount. Such substances may be considered to pass gradually and continuously from the state of fluent liquid, through a condition of continuously increasing viscosity until finally a rigidity comparable with that of ordinary crystalline substances is attained, though the substance is still, from the point of view of the physical chemist, an undercooled liquid. The substance is then said to be in a vitreous condition or is called, simply, a glass.

Silicates are the best known representatives of this class of substances that can be readily undercooled to a vitreous condition, though there are many more or less related substances that behave similarly, such as borates, and still others wholly unrelated, such as ordinary ethyl alcohol. Indeed, the difference between various substances in this respect is only a matter of degree, and it is probable that all substances could be obtained in a vitreous state if the requisite conditions of cooling could be attained.

The conditions that favor permanent undercooling will be apparent from the following considerations. Above the melting point the tendency of a substance to crystallize is zero; at the melting point it is still zero, but as the liquid is cooled below this point the tendency increases rapidly and if there were no counter effect it would probably increase indefinitely as undercooling is increased. However, as the liquid cools there is a constant diminu-

tion in the freedom with which the atoms can arrange themselves in that configuration characterizing the crystals, because the viscosity or internal friction of the liquid continually increases. The increasing viscosity therefore works against the crystallization tendency and the result is that the actual crystallizing power usually increases rapidly as cooling proceeds below the melting point, passes through a maximum, and falls away practically to zero.

The crystallizing power may be defined as the numbers of centers of crystallization established per unit of volume in unit time. From the considerations outlined above it is apparent that its variation with temperature can be represented by curves of the form shown in Fig. 1. Curve A is that of a substance whose crystallization power increases very rapidly when the melting point is passed and attains a very great value. It represents a substance that is very difficult to cool quickly enough to prevent the formation of numerous centers of crystallization. Curve B is that of a substance in which it is difficult to avoid crystallization, but in this case it is not so much because the absolute value of the crystallizing power is so great at any given amount of undercooling, but because the crystallizing power is comparatively great through a long range of undercooling. Hence it is difficult to cool through this long range at a rate sufficiently great to prevent crystallization. Curve C is that of a substance easily undercooled without crystallizing.

Silicates do not all belong to the class of substances represented by curve C. Compounds such as $MgSiO_3$, Al_2SiO_5, and $CaSiO_3$ belong rather to the class of substances represented by curve B. They, therefore, can be cooled to the vitreous state only when the rate of cooling is made very great. On the other hand, a large number of silicates, particularly those of the alkalis, can be cooled quite slowly without crystallization and therefore might be represented by curve C. Nevertheless they can be crystallized with sufficiently slow cooling.

Since molten silicates have comparatively great heat capacities and are poor conductors of heat, it is not possible to cool a large mass at any very rapid rate. Silicates that crystallize readily, such as $CaSiO_3$, can therefore be cooled to the glassy con-

dition only in a very small mass, usually a few grams. Such silicates cannot, therefore, be used as ordinary commercial glasses, though they may enter into the composition of such glasses because in mixtures their specific properties are modified.

Before passing to the consideration of mixtures, however, it is desirable to consider the heating of an undercooled liquid or

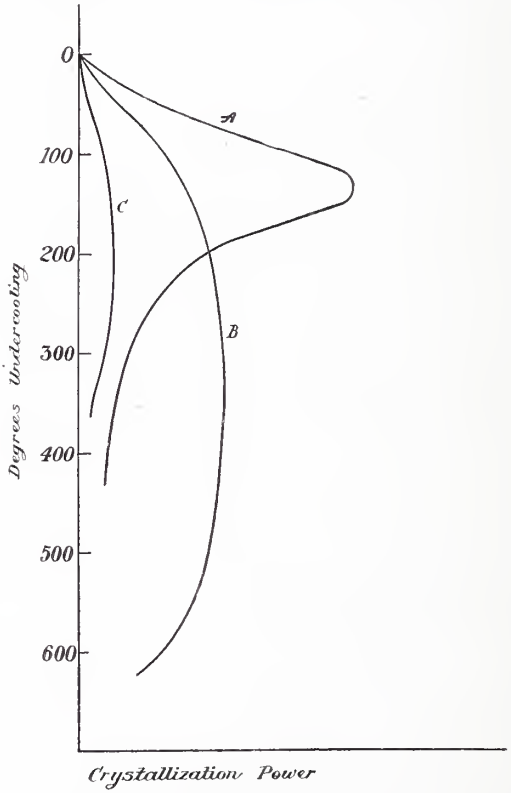

FIG. 1.—Types of crystallization curves.

glass, that is, the reversal of the process of cooling that has been discussed in the foregoing. When such a substance is heated the behavior is the exact reversal of that involved in undercooling. The rigid glass gradually softens, becomes a viscous liquid, and finally a fluent liquid without discontinuity of behavior at

any temperature. Properly speaking, then, a glass has no melting point.

The curves that were given to represent crystallizing power for the liquid which is being undercooled, substantially represent crystallizing power for the reheated glass also. If the substance is of the class of substances represented by curve B, then it must be heated exceedingly rapidly to a temperature above the melting point of the crystalline substance in order to prevent crystallization. Glass of the composition $CaSiO_3$, for example, will crystallize in a short time if held at a temperature even several hundred degrees below the melting point of the crystalline material, and if raised to a temperature not far below the melting point, its crystallization is practically instantaneous. If the behavior of the substance is represented by curve C, on the other hand, then it may be heated slowly and maintained for comparatively long periods at temperatures not far below the melting point of the crystalline substance without crystallization occurring.

In the foregoing, consideration has been given only to the behavior of pure definite compounds. When we pass to mixtures of compounds, as represented in ordinary commercial glasses, we find that certain other factors must be considered. The principles involved in the crystallization of mixtures may be illustrated with the aid of Fig. 2, which represents the crystallization temperatures of all mixtures of $CaSiO_3$ and SiO_2. From the figure it is apparent that, while the crystallization temperature of pure $CaSiO_3$, under equilibrium conditions, is 1540° C, when mixed with 15 per cent SiO_2, its crystallization temperature is lowered to 1490° C, and this is merely the temperature at which crystallization begins. Unlike the pure compound $CaSiO_3$, this mixture does not crystallize entirely at a definite temperature, but as the temperature falls crystallization of $CaSiO_3$ continues and the remaining liquid therefore changes its composition until finally at 1435° C, when the liquid has the composition 78 per cent $CaSiO_3$, the silica begins to crystallize and the whole mass solidifies at this temperature, giving a mixture of $CaSiO_3$ and silica. Such is the behavior when equilibrium prevails, but just as in the case of pure compounds, so in mixtures, undercooling may occur and for similar reasons. Thus the crystallizing ten-

dency of $CaSiO_3$ from the mixture just discussed is zero at all temperatures above 1490° C, while as cooling proceeds below this temperature the tendency increases rapidly, but the counter effect of increasing viscosity enters in so that the actual crystallizing power is never very great. Cooling to a glass is therefore quite readily accomplished with this mixture though so difficult with the pure compound $CaSiO_3$. The effect of the addition of silica is to lower the temperature of crystallization of $CaSiO_3$ to a tem-

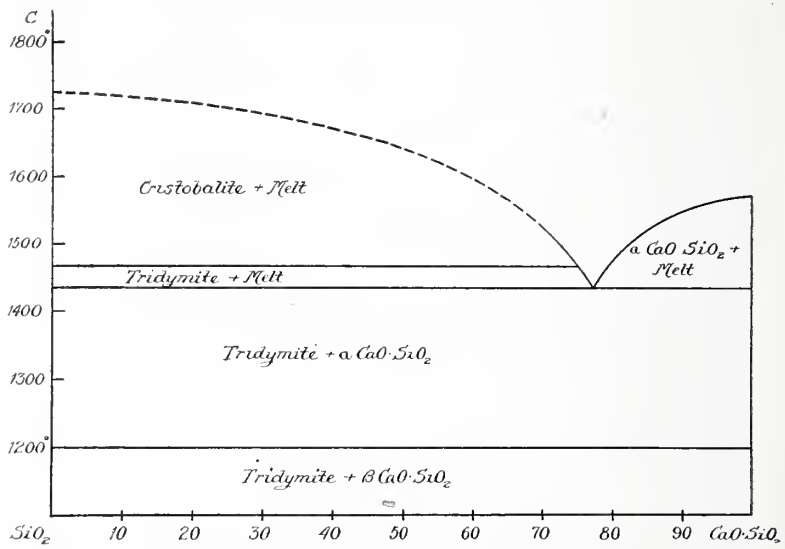

FIG. 2.—Temperature-composition diagram for the system calcium metasilicate–silica ($CaSiO_3$–SiO_2).

perature at which the viscosity is much greater and therefore the crystallization power much decreased. At the same time the silica in virtue of its own inherent properties makes a further addition to the viscosity of the liquid. We thus see that a substance that has a great crystallizing power when pure may nevertheless not separate readily from mixtures, and in general its tendency to separate will be in direct relation to its proportion in the liquid.

In speaking of the cooling of this mixture (85 per cent $CaSiO_3$, 15 per cent SiO_2) it has been stated that below 1490° C the liquid is undercooled. Since we are dealing with a mixture containing

more than one substance we may apply the conception of solutions and the liquid may be said to be supersaturated with respect to $CaSiO_3$ at all temperatures below 1490° C. At 1490° C it is just saturated, and above that temperature it is unsaturated, with respect to $CaSiO_3$. The statement that a certain glass is supersaturated with a certain compound is, therefore, entirely without meaning unless a temperature is specified. Ordinary glasses at room temperature are supersaturated with respect to several compounds, as we shall see in the following.

It has been shown that from the mixture whose crystallization was discussed, crystallization of $CaSiO_3$ began at 1490° C and was continued down to 1435° C when crystallization of silica began, assuming equilibrium. If undercooling occurs, however, the liquid becomes undercooled or supersaturated with respect to $CaSiO_3$ at 1490° C, and undercooled or supersaturated at 1435° C with respect to silica also. Now for each of these compounds there will be a curve of crystallization power, analogous to the curves representing that property that have already been mentioned, though differing, of course, from the curves for the pure compounds in the absolute values represented. In cooling a mixture of the composition discussed, the rate must be made such that the glass does not remain long within the temperature range where the power of crystallization of $CaSiO_3$ or SiO_2, or both, is great. In practice this means that the glass must be cooled rapidly to a temperature at which the crystallization power of these substances is negligible. Similarly, in reheating the glass the temperature should not be raised above that at which their crystallizing power is negligible, or if this is done the time during which it is held above this temperature must be brief, otherwise $CaSiO_3$ or SiO_2, or both, may crystallize out.

When we pass to mixtures of more than two components the same general principles hold, but in this case we have, on cooling, a certain temperature at which the liquid becomes saturated with one of the components, the so-called primary phase, a lower temperature at which it becomes saturated with the secondary phase, and so on, for all the components. If cooling is so conducted, the glass may become undercooled or supersaturated with respect to these various phases at the respective temperatures.

On reheating such a poly-component glass any or all of the components may separate if the necessary relation between crystallizing power and duration of heating is realized.

If we regard the process of manufacture of glass in the light of the foregoing discussion of the theory of devitrification, we find that there are three distinct danger periods during which devitrification is liable to occur. At the full melting or fining temperature no trouble need be anticipated, but when the glass is cooled preparatory to working, some devitrification may occur. Whether the cooling be for the purpose of gathering from pots or from the gathering end of a tank, for the purpose of casting in the case of plate glass or with the object of giving the final stirring before setting out in the case of optical glass, the same considerations apply. The temperature to which the glass is cooled and at which it is usually maintained for long periods must not be such that the glass becomes supersaturated with respect to any of its components; or, stated in another form, the composition of the glass must not be such that it becomes supersaturated with any of the components at the temperature at which it is to be maintained. When devitrification or crystallization does occur at the comparatively high temperature with which we are here concerned, it usually takes the form of the separation of large individual crystals, for the glass is fairly fluid and actual equilibrium conditions may prevail or a sufficiently close approach thereto to allow the crystals to grow slowly by diffusion without the precipitation of the myriad tiny crystals that form when the glass is colder. A particularly good case of this type of devitrification fell under the writer's experience and will be described rather fully here on account of the principles it illustrates and because it affords an example of the application of scientific methods to glass problems.

In the manufacture of the variety of optical glass known as light barium crown much trouble was experienced from the formation of crystals that ruined the greater part of the pot of glass. These crystals formed during the final stages of stirring, immediately preceding the time at which the pot was set out for rapid cooling. The most obvious remedy and the one first tried was to set the pot out at a temperature above that at which the crystals form, and permit the glass to cool more rapidly through

the dangerous temperature. This procedure was entirely successful in eliminating crystallization, but was found undesirable for another reason, *viz.*, that the final stirring of the glass should be conducted at a certain fluidity, and if the pot were set out at a higher temperature then the fluidity of the glass during the final stirring was too great. It was then necessary to attack the problem in another way, and from the principles that have been discussed in an earlier part of the paper it is obvious that if the temperature of final stirring could not be raised then the composition of the glass must be changed so that separation of the crystals would not occur at the temperature normally used. The composition could, of course, have been varied by the method of cut and try, but in this instance the method was adopted of determining the nature of the crystals and deducing therefrom what the change of composition should be. The crystals were very thin hexagonoid plates that probably could not have been identified by purely chemical means, but by determination of optical properties they were identified with the compound $BaSi_2O_5$.[1] Evidently, then, the glass, at the temperature concerned, was supersaturated with $BaSi_2O_5$ and crystallization of that compound could be avoided by lessening the barium content of the glass. At the same time it was desirable to make as little change as possible in the optical properties of the glass so the following method was adopted of determining the minimum amount of decrease in the barium content that would produce the desired effect: The melting point of pure $BaSi_2O_5$ was determined and found to be 1426° C, and the temperature at which the light barium crown glass became just saturated with $BaSi_2O_5$ was determined and found to be 1100° C. The composition of the glass was such that it contained 57 per cent $BaSi_2O_5$, that is, a change of 43 per cent (100–57) lowers the temperature of saturation 326° C (1426–1100). If, therefore, we regard the variation of the temperature of saturation as linear, each further decrease of 1 per cent should lower the temperature of saturation about 7.5° C. This assumption of a linear relation gives, however, only a minimum estimate, because all curves of this nature for silicates

[1] N. L. Bowen, "Crystals of Barium Disilicate in Optical Glass," *J. Wash. Acad. Sci.*, **8**, 265 (1918).

that have been investigated are concave against the temperature axis (see Fig. 2) so that the curve would have the form shown in Fig. 3 rather than the straight line also shown in that figure. A fairly safe assumption is that the curve would have at

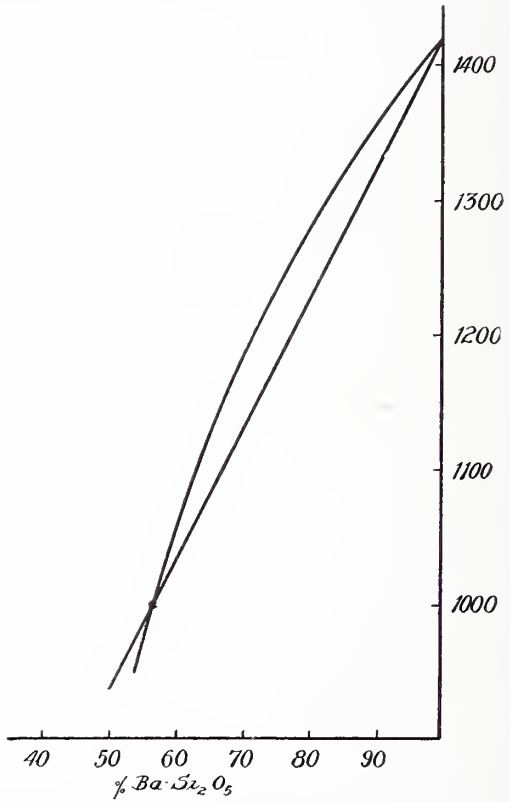

FIG. 3.—Diagram to illustrate the probable form of the curve of saturation temperature of a barium crown glass with respect to barium disilicate.

the point in question about twice the gradient of the straight line and therefore a change of 1 per cent $BaSi_2O_5$ should make a change of about 15° C in the temperature of saturation. Experience had shown that the actual separation of these crystals occurred at a temperature about 70° C above that at which it was desirable to conduct the final stirring. A change of composition

sufficient to reduce the temperature of saturation about 70° C, or a little more, should therefore be ample to permit final stirring at the desired temperature without danger of separation of $BaSi_2O_5$. From the above considerations it is plain that the change of composition should be such as to reduce the $BaSi_2O_5$ content about 5 per cent.[1] Accordingly this change was made and the result was all that could be desired. Thereafter no rejection of light barium crown glass on account of the presence of crystals was necessary.

The case of the separation of $BaSi_2O_5$ is the only example, in the writer's direct experience, of the crystallization of a glass at a comparatively high temperature at which the individual crystals were enabled to grow to comparatively large dimensions. The writer has had submitted to him, however, a sample of plate glass containing crystals of wollastonite ($CaSiO_3$) up to 4 mm. in length that formed in the pot during the "hold-over" immediately preceding the casting of the plate. During casting, the crystals all became arranged with their longer axes parallel to the direction of rolling. The exact conditions under which these crystals formed are not available, but an analysis of the glass, kindly furnished by Mr. F. Gelstharp, shows it to contain a slightly higher amount of lime than normal plate. Whether this slight excess of lime is sufficient to render the glass supersaturated with $CaSiO_3$ at the normal casting temperature is a question. Whether the casting temperature was lower than normal the writer was unable to ascertain, but it seems probable that a combination of these two factors entered into the conditions controlling the separation of the crystals.

Le Chatelier describes some very large crystals of tridymite (SiO_2) that formed in a flint glass that was unintentionally held at a temperature about 800° C for a long period.[2] These crystals evidently grew rather freely under conditions approaching equilibrium and tridymite is the primary phase for glass of that composition. The same glass can be held for moderate periods at

[1] It should be noted that deductions as to changes of composition that will produce a specific result can be regarded as reliable only for small changes close to a known point.

[2] *Bull. Soc. Min. Fr.*, **39** (1916).

800° C without danger of crystallization, so the separation of crystals in the case cited illustrates the importance of the time factor.

Another danger period in the manipulation of glass is that during which it is cooled from its final temperature in the furnace or tank, when it is plastic, to lower temperatures at which it is rigid. During this period it is usually worked into form whether by blowing, drawing, casting, or otherwise, but in the case of optical glass the "metal" is usually cooled in the pot itself. Since the rate at which a large mass of glass can be cooled is limited on account of the low conductivity of glass, the cooling of glass as a large mass in the pot is likely to bring about devitrification. Under these conditions devitrification never takes the form of the separation of large individual crystals but may appear in the form of spherulites (spheroidal aggregates of crystals with a radiating fibrous structure), or in the form of very small crystals evenly disseminated. This latter form of devitrification may give an opalescent glass, a milky glass, or a dense, opaque glass, according to the size of the crystalline particles that separate. In opalescent or milky glass the crystalline particles are beyond the resolving power of the microscope but can readily be detected with the ultramicroscope. In a dense, opaque, devitrified glass the particles can be seen by the microscope and their refraction relative to that of the glass observed. In the writer's experience the crystals are apparently, in nearly all cases, one of the low-refracting forms of silica, namely tridymite or cristobalite. Certain substances, which, for want of a better term, may be called mineralizers, seem to exert an influence in promoting this form of crystallization. Among these are SO_3 and Cl.[1] Arsenic, also, when present in excess of certain amounts, acts similarly, and in one case in the writer's experience the devitrification of a medium flint (n_D = 1.62) was eliminated by cutting down the arsenic in the batch from about 0.9 to about 0.3 per cent. Fluorine probably acts in a similar capacity and certain glasses that are deliberately rendered opaque by the addition of fluorides no doubt owe their opacity (crystallization)

[1] Fenner and Ferguson, "The Effect of Certain Impurities in Causing Milkiness in Optical Glass," *J. Am. Ceram. Soc.*, 1, 468 (1918).

to a separation of crystals that is aided by fluorine. In some cases fluorides themselves may represent the crystalline phase. On the whole, however, little is known of the fundamental facts concerning these opaque and translucent glasses.

More definitely susceptible of solution, however, are the problems connected with the other form of crystallization that takes place during this period of cooling of the glass now being considered. The crystalline constituents of spherulites are readily determinable under the microscope, and, if the heat treatment cannot be varied in such a way as to eliminate their formation, a knowledge of the composition of the crystals enables one to make appropriate changes in the composition of the glass.

The type of barium crown glass whose devitrification at high temperatures has already been described, may further devitrify during the cooling stage with separation of spherulites of $BaSi_2O_5$ that grow about the larger crystals of earlier separation, or with the formation of new simple spherulites. The remedy for this is either (1) the reduction of the $BaSi_2O_5$ content of the glass, just as at the higher temperature (see page 269), or, (2) more rapid cooling. Moreover, even barium crown glass that is safe from devitrification at this stage, in so far as the main mass of glass is concerned, may nevertheless devitrify at the free upper surface of the glass with the formation of spherulites of $BaSi_2O_5$.

From ordinary crown glasses that have an excess of lime as compared with fluxes, spherulites of wollastonite, $CaSiO_3$, sometimes separate during the cooling stage.

Some types of borosilicate glasses may devitrify with separation of spherulites of tridymite, SiO_2. A remedy for this in one instance was found to be the substitution of potash for some of the soda of the glass, a change that probably had the effect of increasing the viscosity and thus diminishing the crystallizing power at the temperature concerned.

The final period of danger of devitrification is one of considerable importance. It is that period during which the glass is reheated, whether for working it into a desired form or for annealing it if it is already in final form. The interrelation of time, temperature, composition and power of crystallization must be carefully borne in mind.

If the glass is to be worked into shape it must, of course, be

heated to a temperature such that it is subject to flow. In the case of optical glass, for example, a common procedure is to heat in molds the rough chunks from the broken-down pot, the operation being conducted at such a temperature that the glass sags down and takes the shape of the mold. There is a certain amount of choice of temperature available, for obviously the sagging will proceed at a certain rate at a certain temperature and at a greater rate at higher temperatures. In the discussion of the principles underlying crystallization it was pointed out that for any glass there is a certain temperature below which the crystallization power is negligible, and another temperature above which the crystallizing power is zero. The lower limit has no special significance in connection with the molding process, for all glasses are too rigid for molding at these temperatures. The upper limit, however, has great importance for if the molding is conducted at a temperature slightly higher than this upper limit there is absolute certainty of freedom from devitrification during the molding itself, though subsequently, of course, proper precautions must be taken to cool at an adequate rate. It is highly desirable, therefore, to know the temperature of this upper limit of devitrification for individual glasses. This temperature is, it will be recalled, that at which the primary phase begins to separate when cooling of the glass is carried on under perfect equilibrium conditions. It is identical with the temperature at which the crystallized glass becomes completely molten as the temperature is raised. This fact can be used in order to determine the temperature of this upper limit. A small piece of devitrified glass is held at a certain temperature for about an hour, is rapidly chilled and examined, this operation being repeated at successively higher temperatures until one is reached at which the last traces of crystals have disappeared. Unless it is undesirable for other reasons, such as the development of bubbles in the glass, molding should be conducted at a temperature slightly higher than that determined by the above method, absolute freedom from devitrification during the molding process itself being thereby guaranteed. Molding can be conducted at lower temperatures with some glasses whose devitrification tendency is small.

The temperatures found for various glasses examined in connection with optical glass investigation are shown in Table 1.

TABLE 1.

Data on Devitrification of Some Optical Glasses.

Glass.	Temperature of disappearance of crystals.		Composition of crystals. (Primary phase.)
Medium Flint (n_D = 1.62).....	910° C.	1670° F.	SiO_2
Ordinary Crown.....	1110° C.	2030° F.	SiO_2
Light Barium Crown.....	1100° C.	2012° F.	$BaSi_2O_5$
Light Flint.....	1115° C.	2039° F.	SiO_2

Glass is frequently reheated not alone for working but also for annealing. All glass must be free from excessive strain that is likely to cause breakage, and optical glass in particular must have the strain reduced below the quite moderate amount that will cause a warping of reflecting or refracting surfaces during grinding and polishing; in short, optical glass must be subjected to fine-annealing. In annealing, the glass is raised to a temperature at which the strain is relieved by relatively slow flow. Obviously there is again a certain amount of choice of temperature permissible, for the flow will take place slowly at low temperatures and more rapidly at higher temperatures. It is important, therefore, to know the crystallizing power for various temperatures of the glasses to be annealed, in order that a temperature may be chosen that will not induce devitrification. The choice is not so easily made as it was for molding purposes, for there is no definite lower limit of devitrification temperatures analogous to the upper limit that has been pointed out, and while there is for any glass a practical lower limit below which the power of crystallization or devitrification is negligible, this is, unfortunately, in the case of some glasses, a temperature at which rate of annealing is of like magnitude. At low temperatures devitrification is induced in a certain time at a certain temperature, in a shorter time at a higher temperature, and so on. It is

therefore desirable to know for any glass whether devitrification will be induced at any given temperature in a period of time which

TABLE 2.

Experiments on Devitrification of Certain Optical Glasses.[1]

Time. Hours.	Temperature °C.	Temperature °F.	Results.
	Barium Flint Glass.		
15.0	660	1220	No devitrification.
27.5	700	1292	No devitrification.
29.0	750	1382	No devitrification.
68.0	800	1472	No devitrification.
	Medium Flint Glass (n_D = 1.62) Crystals; SiO_2.		
1.0	650	1202	No devitrification.
12.5	650	1202	Slight surface film.
4.5	700	1292	No devitrification.
6.5	700	1292	Slight surface film.
23.5	700	1292	Marked surface film.
	Borosilicate Crown Glass. Crystals; SiO_2.		
14.5	660	1220	No devitrification.
27.5	700	1292	Surface devitrification.
4.5	750	1382	Surface devitrification.
8.0	750	1382	Surface devitrification.
2.3	800	1472	Surface devitrification.
20.6	800	1472	Surface devitrification.
	Ordinary Crown Glass. Crystals; SiO_2 and $CaSiO_3$.		
41.0	600	1112	No devitrification.
36.0	650	1202	No devitrification.
4.0	700	1292	Slight surface film.
6.5	700	1292	Surface film.
23.5	700	1292	Thick surface layer.
33.0	700	1292	Thick surface layer.
47.0	700	1292	Thick surface layer.

is long enough to give adequate annealing at that temperature. A fore-knowledge of both rates of devitrification and of rates of

[1] Temperature measurements by J. C. Hostetter. Identification of crystalline phases by N. L. Bowen.

annealing for various glasses is therefore requisite. It is plain, too, from the foregoing discussion, that glasses will be encountered in which fine-annealing and freedom from devitrification are mutually incompatible.[1] In such a glass every effort should be made to change the composition in such a manner as to increase the annealing rate or decrease the devitrification rate, or to accomplish both these results simultaneously. In making a change of composition a knowledge of the nature of the phases that separate is again desirable, precisely as it was for devitrification at other stages in the manipulation of the glass. Devitrification during annealing may take the form of the separation of spherulites or of minute crystals more evenly distributed that give a milky or dense, opaque glass. The identification of the crystals in the spherulites is usually readily accomplished and in the opaque glass one can frequently obtain a clue as to the nature of the crystals by observation of the relative refraction of crystals and glass. In milky glass one cannot determine the crystals, but intensification of the effect can frequently be obtained by raising a piece of the glass to a somewhat higher temperature. The larger crystals thus formed may then be identifiable.

In Table 2 the results are given of a study of devitrification of various glasses at these lower temperatures. It should be remarked that frequently the devitrification may take place only at the free surface of a piece of glass, but the reason for this greater freedom of devitrification at the surface is unknown.

Summary.

Devitrification of glass is the result of the tendency of the glass to reach the stable crystalline condition, and takes place whenever the glass is held for a sufficiently long period of time within the range of temperature where its crystallizing power is great. The various forms of devitrification in glass are discussed from this point of view and suggestions are made as to the principles that must be borne in mind in deciding upon modifications of

[1] It is perhaps worthy of note that certain opaque (devitrified) glasses that are used for ornamental purposes are clear when first cooled to the desired shape and become opaque only on reheating which accomplishes the double purpose of annealing and devitrification.

procedure or changes in composition that have as their object the avoidance of devitrification. Specific examples of the devitrification of optical glasses are given, together with determinations of the temperatures of devitrification and the crystalline phases separating.

GEOPHYSICAL LABORATORY,
CARNEGIE INSTITUTION OF WASHINGTON.
QUEEN'S UNIVERSITY, KINGSTON, ONTARIO,
February, 1919.

COMMUNICATED DISCUSSIONS.

W. S. WILLIAMS: The thanks of the glass technologists are due Dr. Bowen for his able exposition of some of the fundamental principles of physical chemistry applied to glass. It is to be regretted that the compositions of the glasses which devitrified are not given. They would serve as a guide and a warning in the manufacture of these types of glasses.

In my experience in the manufacture of optical glass at the Bureau of Standards, I have never experienced devitrification of light barium crown, $n_D' = 1.57$. All of the compositions used were compounded with a knowledge of ceramic chemistry and will be published in this JOURNAL. Of these, only one type tends to devitrify when the technic described in the above article is followed. This is the dense barium crown, $n_D = 1.60$. I have seen only two melts of this type devitrify during the melting stage. The composition of the first one was

	Per cent.
SiO_2	40.7
B_2O_3	6.2
ZnO	9.3
BaO	43.2
As_2O_3	0.6
	100.0

This batch was filled in sixteen hours at 1350° C and was closed and held at 1350° C for eight hours, during a fining period. When a proof was taken a slight scum was observed and skimmed off. Two hours later, however, a scum about 3 inches deep had formed.

This was also skimmed and about three inches of clear glass was taken out of the pot with the purpose in view of endeavoring to eliminate the nuclei, if possible. The temperature was raised 50° C to 1400° C. Three hours later, however, fully 6 inches of glass showed devitrification and the melt was abandoned. When cold the melt exhibited about 12 inches of dense, pure white opal glass on the surface and the rest of the glass was clear. Chemical analyses of the clear glass and opal glass demonstrated marked differences in their compositions. The analyses were as follows:

	Clear glass.	Opal glass.
SiO_2	37.88	44.67
BaO	42.36	38.02
ZnO	10.52	9.25
B_2O_3	9.31	8.46

A petrographic examination was baffling—due to the extremely fine state of the crystals. This was a case of devitrification occurring during melting and fining. In subsequent melts the difficulty was eliminated by the addition of Al_2O_3.

The second case of devitrification in dense barium crown, $n_D = 1.60$, occurred with a batch of the following composition:

	Per cent.
SiO_2	37.0
B_2O_3	5.0
K_2O	2.7
ZnO	7.8
BaO	47.0
As_2O_3	0.5

This batch was filled in 15 hours at 1400° C and fined for two hours. During a stirring period of 12 hours the temperature was gradually reduced to 1100° C. No devitrification was observed, but when the melt was cold spherulites were seen on only one side of the pot about 8 inches down from the surface. They extended about 6 inches from the side of the pot into the glass. These spherulites were determined under the microscope to be barium disilicate. With the exception of these few nodules the glass was good.

During molding and annealing dense barium crown, $n_D = 1.60$, of all compositions, devitrifies readily and must be molded in the shortest possible time. Sticking on a punty and heating in a glory hole is the most satisfactory method of molding, as the heat is more intense and the glass more uniformly heated than by other methods.

Bureau of Standards,
Pittsburgh, Pa.

E. W. Tillotson: In this paper Mr. Bowen has made a noteworthy contribution to the subject of devitrification. As a natural consequence of his observations of the separation of crystalline material, mention is made in several places of the relation of devitrification to the production of translucent and opaque glasses. The writer would therefore like to inquire whether Dr. Bowen would exclude as possible causes of turbidity in glass the formation of "emulsions"[1] (glass in glass) or of "foams" (gas in glass).

Mellon Institute of Industrial Research,
Pittsburgh, Pa.

N. L. Bowen: The compositions of the glasses on which we made devitrification experiments were not given in the article, because the original batches of Table 2, and of the ordinary crown and light barium crown in Table 1, had been used by the chemists of the Pittsburgh Plate Glass Company before the dat when the party from the Geophysical Laboratory was invited to cooperate in optical glass manufacture at the Charleroi plant. The officials of the Company have requested that these compositions should not be published. Changes were subsequently made in the formulas of some of these glasses, particularly in the light barium crown, which, on account of devitrification, was not satisfactory until its composition had been altered as indicated on page 271.

I have seen a sample of a scum formed on barium glass similar to that described by Mr. Williams though I was not present during the making of the melt itself. Under the microscope this scum was found to consist of glass with a cloud

[1] See for example, Guertler *Z. anorg. Chem.*, **40**, 225 (1904).

of minute crystals of silica in one of its low refracting forms, tridymite or cristobalite.

Evidently Mr. Williams' scum was identical, for a simple calculation shows that if one took 9 grams of material corresponding in composition with his "Clear Glass" and added thereto 1 gram of silica, one would obtain a material of the composition of his "Opal Glass." The continued formation of this scum as observed by Mr. Williams was therefore the result of the rising of these minute crystals of silica towards the surface. Whether the formation of these crystals is a true example of devitrification is questionable; indeed, it seems rather probable that they may represent completely recrystallized silica that had never completely dissolved because the melt was too slow. The fact that undissolved silica will rise through the glass in this manner is clearly demonstrated by Fig. 1 in my paper on "Identification of Stones."[1]

In reply to Dr. Tillotson, I would say that formation of emulsions and foams should not be excluded as possible causes of turbidity in glass, but in the turbid glasses that I have seen there seems little reason to believe that anything other than crystalline particles are the underlying cause. One can usually modify the treatment that renders a glass turbid in such a way (for instance, by prolonging the treatment) that the particles causing turbidity become sufficiently large for identification as crystal particles.

[1] This Journal, 1, 598 (1918).

SOME PHYSICAL PROPERTIES OF AMERICAN COMMERCIAL PORCELAIN BODIES.[1]

By J. W. Wright and S. I. Sewell.

Introduction.

The purpose of presenting this work is to make possible the comparison of the physical properties of several commercial porcelain bodies used in the manufacture of table ware. It is desirable to have available physical constants relating to the mechanical strength and thermal properties of porcelain bodies; first, in order to establish data of general interest; second, in order to facilitate the replacing of materials now used by others. Such data is necessary if domestic clays are to be used in replacing those which are imported.

The work completed to the present time has afforded the following data for purposes of comparison:

1. Water content in the plastic state.
2. Drying shrinkage by volume.
3. Transverse strength in the dry state.
4. Compressive strength in the dry state.
5. Compressive strength in the fired state.
6. Vitrification behavior.

The Investigation.

Six commercial porcelain bodies were secured from potteries and numbered C-1, C-2, C-3, C-4, C-5 and C-8, respectively. These bodies were received in the plastic state. Although they had been packed with damp cloths, they were too stiff to work well on reaching the laboratory, except Body C-8, which was delivered the same day it was shipped from the pottery. The other bodies were brought, as near as possible, to the same consistency. Included with these bodies were two prepared in our laboratory. These laboratory mixtures were as follows:

[1] By permission of the Director, Bureau of Standards.

Body No. 163.

		Per cent.
Tenn. No. 9 ball clay		8.0
Georgia kaolin Florida kaolin Delaware kaolin N. Carolina kaolin	1/4 each	37.0
Feldspar		18.0
Flint		37.0

Body No. 175.

Tenn. No. 9 ball clay		5.87
Georgia kaolin Florida kaolin Delaware kaolin N. Carolina kaolin	1/4 each	38.28
Feldspar		17.71
Flint		36.30
Whiting ($CaCO_3$)		0.89
Magnesite ($MgCO_3$)		0.95

Body No. 163 was ground in a ball mill for a period of forty-eight hours, while body No. 175 was ground for only three hours.[1]

1. **Water Content in the Plastic State.**—The values (Table 1) represent the averages of the results on three test specimens and were computed in percentages of the dry weights. The weights of the briquettes were taken immediately after they had been made; they were then dried at 110° C and after cooling in a desiccator the dry weights were determined.

2. **Drying Shrinkage by Volume.**—The values (Table 1) represent the averages of the results on three test specimens and were computed in percentages of the dry volumes. Immediately after the specimens had been made they were immersed in kerosene and the volumes were determined in a volumeter of the Seger type. After drying at 110° C and cooling in a desiccator, the dry volumes were determined in a like manner.

3. **Transverse Strength in the Dry State.**—The values (Table 1) represent the average moduli of rupture in pounds per square inch of ten specimens broken on a transverse-strength testing-machine

[1] Riddle and McDanel, *J. Am. Ceram. Soc.*, 1, 620 (1918).

The specimens for this test were bars, 1″ × 1″ × 7″, shaped by pressing into a brass mold, using a piece of plastic body previously cut by a wire to as near the size of the mold as possible, pressing, and then cutting off the excess with a wire. The bars were dried at 110° C, cooled in a desiccator, and broken, From the dimensions of each specimen and the breaking load, the moduli of rupture were calculated by the use of the usual formula.[1]

4. **Compressive Strength in the Dry State.**—The values (Table 1) represent the averages of determinations on seven specimens, calculated in pounds per square inch. The specimens were $1^1/_8$″ cubes, cut from a plastic bar of clay which had been shaped in a manner similar to that used in making the transverse-strength bars. Before crushing, the test pieces were dried at 110° C, cooled in a desiccator, and two opposite sides made parallel and smooth by rubbing with sand paper.

5. **Compressive Strength in the Fired State.**—The values (Table 1) represent the higher results on one of two specimens and were computed in pounds per square inch. Cylinders for this test were made by forcing a column of clay through a die,

TABLE 1.

Body No.	Pounds per square inch.			Per cent water content.	Per cent volume drying shrinkage.
	Compressive strength in dried state.	Transverse strength in dried state.	Compressive strength in fired state.		
163		112	26,300	34.40	16.73
175		116	39,000	32.17	15.87
C-1	435	174	39,500	23.51	7.96
C-2	509	215	49,500	26.88	10.83
C-3	411	144	42,650	27.37	11.71
C-4	423	170	47,600	28.00	11.90
C-5	495	206	63,500	26.62	11.69
C-8	615	178	40,000	28.90	13.51
Average of the 6 commercial bodies	461	181	47,125	26.88	11.26

[1] Report of the Committee on Standards, *J. Am. Ceram. Soc.*, 1918, 40 (Jan.).

$1\frac{1}{4}$″ in diameter, and cutting the column into three-inch lengths. They were fired to cone 11 and held at the temperature at which this cone went down for a period of four hours. Cone 12 did not show any deformation under this heat treatment. The six commercial bodies showed zero absorption. After grinding the ends of each test cylinder parallel it was crushed in an Olsen testing-machine.

6. Vitrification Behavior.—The porosity and volume changes of the bodies and their firing ranges were determined by means of a draw-trial burn. Briquettes, $1\frac{1}{8}$″ × $1\frac{1}{8}$″ × 2″ were made by forcing a column of the clay through a die and cutting to the desired length. The volumes of the dry briquettes were determined as before. They were placed in a muffle so that one specimen from each body could be drawn at the respective temperatures as shown in Table 2. The deformation of Orton cones at each drawing was also noted. The kiln was fired with natural gas and compressed air under oxidizing conditions. The rate of temperature increase was 20° C per hour for a period of three and one-half hours previous to the first draw and throughout the remainder of the burn. After each set of trials was drawn from the furnace it was placed in an auxiliary furnace, at low-red heat, and at the end of the burn was allowed to cool at the normal rate with the furnace. The dry weights and, after boiling under partial vacuum, the wet and suspended weights were then determined. From these data the porosities were calculated by the use of the Purdy formula.[1] The volumes of the burned pieces were determined and the changes in volume, or the burning shrinkages, were computed in percentages of the volumes of the unburned specimens.

When a body reached zero absorption it was considered matured. The volume shrinkage of a body should approach, but not necessarily reach, the maximum change when zero absorption is first reached. However, maximum shrinkage should not be reached prior to obtaining zero absorption. If a body shows, on reaching zero absorption, a decrease in shrinkage from its maximum, it is likely that the point of over-firing has been

[1] R. C. Purdy, Illinois Geol. Survey, *Bull.* 9.

TABLE 2.

Temperature ° C.		1160.	1180.	1200.	1230·	1260.	1290.	1320.	1350.	1380.	1410.	1440.
Cone deformation.		4 flat.	7 flat.		11 started.	12 3/4 down.	13 1/4 down.	13 flat.		15 down.	16 flat.	17 flat.
Body C-1	% Porosity	18.98	15.10	8.13	7.78	1.76	0.03	0.00	15.25	14.30		
	% Volume burning shrinkage	18.40	21.50	25.00	28.50	30.80	30.82	29.65	18.52	14.82		
Body C-2	% Porosity	25.31	18.45	9.63	5.23	0.08	0.00	0.00	12.65	10.60		
	% Volume burning shrinkage	14.75	20.55	25.10	30.50	33.05	31.80	28.55	21.25	17.00		
Body C-3	% Porosity	27.70	17.85	9.38	4.00	0.00	0.07	18.00	18.80	21.90		
	% Volume burning shrinkage		22.50	28.88	34.55	33.30	25.05	16.73	14.22	10.42		
Body C-4	% Porosity	21.45	16.02	8.86	2.66	0.07	0.07	0.38	14.05	10.42		
	% Volume burning shrinkage	19.95	23.05	27.80	31.70	33.15	31.35	24.65	18.50	14.58		
Body C-5	% Porosity	27.30	20.55	11.57	4.42	0.13	0.04	7.37	11.80	8.77		
	% Volume burning shrinkage	13.59	19.88	26.35	33.00	33.90	30.30	21.92	18.82	15.70		
Body C-8	% Porosity	14.50	9.23	2.47	0.00	0.67	3.35	3.16	12.40	8.32		
	% Volume burning shrinkage	23.40	28.03	31.40	33.25	30.25	20.35	19.25	16.70	15.02		
Body 163	% Porosity			15.82	10.40	0.04	0.00	0.00	0.00	0.00	4.15	1.10
	% Volume burning shrinkage			25.20	30.50	35.00	36.55	36.00	36.30	33.12	22.87	23.05
	% Porosity			8.00	6.57	2.56	0.56	0.04	0.08	0.07	2.35	4.50
Body 175	% Volume burning shrink-											

reached. The volume changes give, undoubtedly, better indications of the firing range of a body—since they indicate over-burning in a much more decisive manner than do the porosity changes. Bodies usually show a tendency to expand before an increase in porosity is observed.

Summary of Results.

The data shown in Table 2 are plotted as curves (Figs. 1, 2 and 3) which can be studied to better advantage. Each diagram shows two commercial bodies in comparison with the two labora-

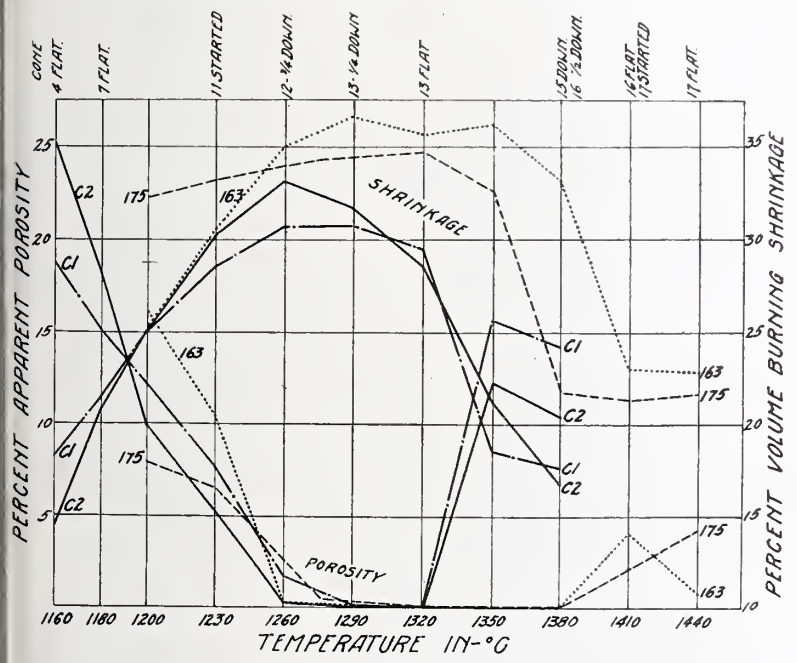

FIG. 1.

tory bodies. Body No. 163 was fired in the same draw-trial burn with the commercial bodies. Body No. 175 had been burned previously, but under similar conditions.

Body C-1 (Fig. 1) reaches maximum shrinkage and approaches zero absorption at 1260° C, or the equivalent to a heat treatment of cone 12, $^3/_4$ down. Over-burning of this body is not in evi-

dence at or below a temperature of 1320° C, thus showing it to possess a firing range extending over an interval of 60° C.

Body C-2 (Fig. 1), like Body C-1, reaches maximum shrinkage and approaches zero absorption at 1260° C. The test pieces show a slight expansion in the two following draws. The firing range can be considered to extend over a temperature interval of 60° C.

The laboratory bodies, Nos. 163 and 175, reached zero absorp-

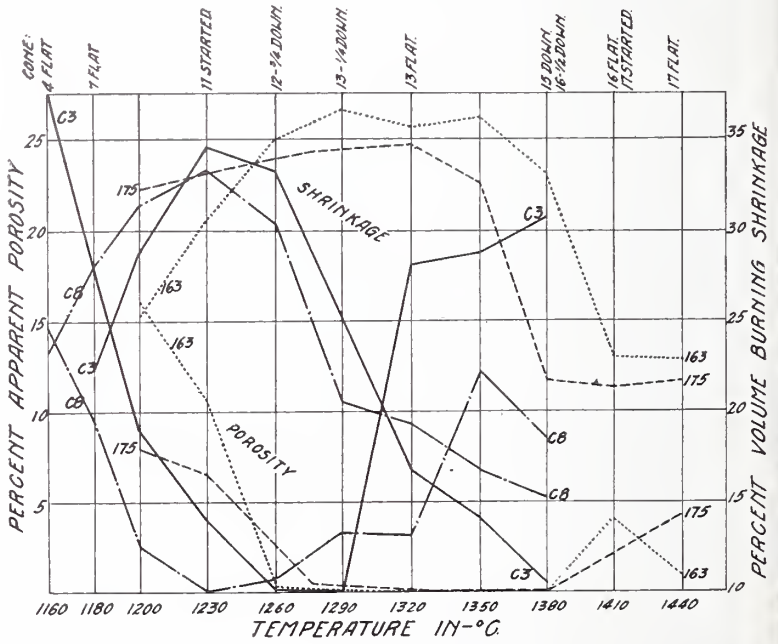

FIG. 2.

tion and approached maximum shrinkage at 1260° C and 1275° C, respectively. The shrinkage and porosity curves show that these bodies have much longer firing or maturing ranges than the commercial bodies. Upon comparing the shrinkage curves, body No. 175 is found to show over-burning before body No. 163. This is to be expected because of the greater content of alkaline earth fluxes of body No. 175.

Body C-3 (Fig. 2) develops its maximum shrinkage at 1230° C

(cone 11 started), but zero absorption is not obtained until 1260° C is reached, whence swelling has begun. At 1290° C, the body still shows zero porosity, but the swelling has been excessive. The firing range of this body is very short.

Body C-4 (Fig. 3) shows maximum shrinkage and zero absorption at 1260° C. At 1290° C a slight but not excessive expansion

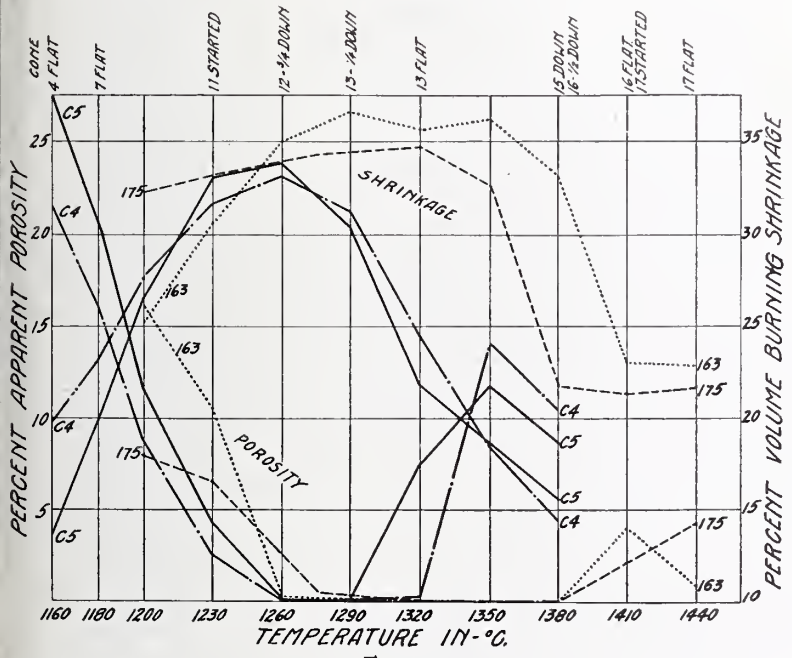

Fig. 3.

occurs. The burning range of this body, according to the draw-trial burn, is approximately 30° C.

Body C-5 (Fig. 3) shows approximately the same burning characteristics as body C-4.

Body C-8 (Fig. 2) shows burning maturity at a lower temperature than any of the other bodies. According to the draw-trial burn, maturity is reached at 1230° C, or the equivalent to a heat treatment of cone 11 started. It shows expansion and an increase in porosity at the next draw, 1260° C. The body has a very short firing range.

The fact that all of the commercial bodies had zero absorptions at cone 11 in the longer heat treatment and not until cone 12 was $^3/_4$ down in the draw-trial burn, in all cases except with C-8, which reached non-absorbent vitrification at cone 11 started, may be attributed to the difference in the rate of temperature increase and its effect upon the fluxes other than feldspar.

Conclusions.

The numerical results obtained in this work are of interest since the determinations were made upon typical commercial bodies which are the result of years of practical experience. They show decided similarity. The strengths of the bodies in the dried state and the strengths of the fired porcelains show no radical differences. There is no evidence that the crushing strength in the dried state is a criterion of the fired strength.

The average figures for the different factors of the six commercial bodies should be of general interest. It will be observed, also, that the water contents and hence the drying shrinkages of the laboratory bodies are higher and the transverse strengths of the dried bodies lower than the corresponding values for the commercial porcelains. Similarly, the compressive strength in the fired state of body No. 163, containing no alkaline-earth fluxes, is the lowest of the series. On the other hand, the temperature ranges at non-absorbent vitrification of the laboratory bodies are longer than those of the pottery bodies—which would indicate the desirability of further work along these lines.

The writers desire to express their thanks to the manufacturers of table-ware porcelain who have coöperated in this work by furnishing the samples of prepared bodies for the tests.

Bureau of Standards,
Pittsburgh, Pa.

EXPERIMENTS IN DEAD-BURNING DOLOMITE.[1]

By H. G. Schurecht.

Introduction.

Magnesite and dolomite are used for linings in furnaces where basic refractories are necessary to resist the corrosive action of basic slags. Some of the uses are as follows:

1. Reverberatory lead-refining furnaces. Lead cupeling-furnaces, where the slag consists chiefly of basic oxides. Crucibles for lead blast-furnaces.
2. Copper converters and copper reverberatory furnaces.
3. Basic open-hearth and Bessemer furnaces.
4. Crucibles for melting metals.

In 1914, 120,583 tons of calcined magnesite[2] were imported into the United States, chiefly from Austria-Hungary. Owing to the war no magnesite was imported into this country in 1916. American manufacturers were, therefore, forced to use California and Canadian magnesites, which, owing to their high degree of purity, were not as satisfactory as the Austrian magnesite—which contains sufficient iron oxide to render it suitable for the manufacture of refractories.

Owing to the fact that dolomite deposits occur nearer to the steel centers, and to its lower cost, experiments were undertaken in dead-burning some Ohio dolomites which are extremely pure and almost as refractory as magnesite.

Some of the difficulties encountered in the use of calcined dolomite for the manufacture of refractory products are as follows:

1. It does not stand storage well. This is due largely to the fact that the raw materials are too pure before calcination—in which case the lime air-slakes readily.

2. It lacks bond when used in a furnace and therefore has a short life. This is also due to the high degree of purity which

[1] By permission of the Director, U. S. Bureau of Mines.

[2] C. G. Yale and H. S. Gale, "Magnesite in 1916," U. S. Geol. Sur. Mineral Resources of the U. S., Part II, pp. 391–401 (1918).

does not permit the dolomite to bond together as it would if some impurities were present.

Previous Work.

A large number of patents have been issued within the past few years on dead-burning the purer grades of magnesite and dolomite.

The methods employed may be arranged in three general groups, as follows:

1. By mixing an impurity[1] with the finely-ground rock and

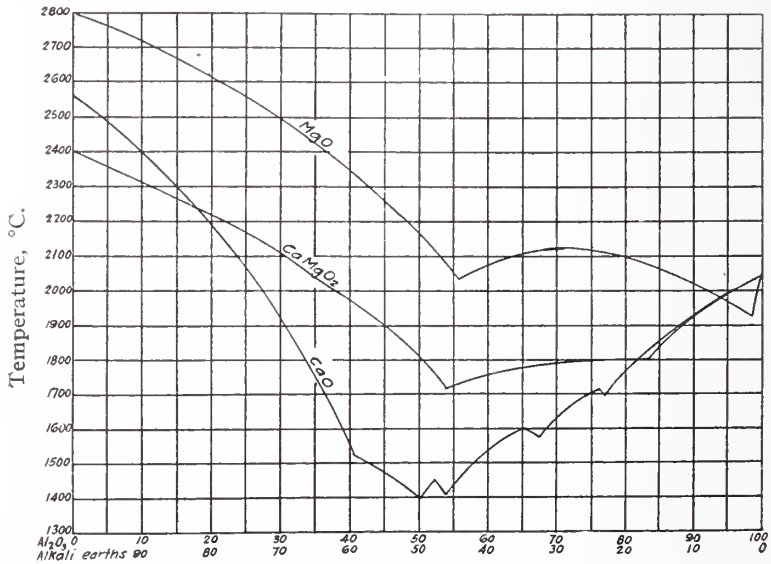

FIG. 1.—The effect of Al_2O_3 on the fusion of CaO, MgO and $CaMgO_2$.

calcining the mixture in a rotary kiln. The impurities recommended are: silica, iron oxide, roll scale, flue dust, alumina, chromium oxide, manganese oxide, cobalt oxide, nickel oxide, borax, calcium chloride, magnesium chloride and sodium chloride.

2. Another method, used by Jones,[2] consists in mixing 90

[1] G. L. Davison, U. S. 792,382, June 20, 1905, "Dead-burning of Magnesite." C. B. Stowe, U. S. 1,205,056, Nov. 14, 1916, "Dead-burning of Magnesite." J. O. Handy and R. M. Isham, U. S. 1,270,818, July 2, 1918, "Dead-burning of Dolomite." S. B. Newberry, U. S. 1,267,686, May 28, 1918, "Dead-burning of Dolomite."

[2] Fred A. Jones, U. S. 151,535, Jan. 1, 1918, "Process of Producing a Furnace Lining with Dolomite."

parts coarsely screened dolomite, 2–8 mesh, with 10 parts flue dust and burning the mixture in a rotary kiln. This product is then used for lining furnaces.

3. The third method used is to separate the MgO from dolomite, $MgCa(CO_3)_2$. It has been found[1] that MgO can be more easily dead-burned than CaO—owing to the fact that CaO has a higher tendency to combine with water and slake. This separation is accomplished in two different ways: one method[2] is by

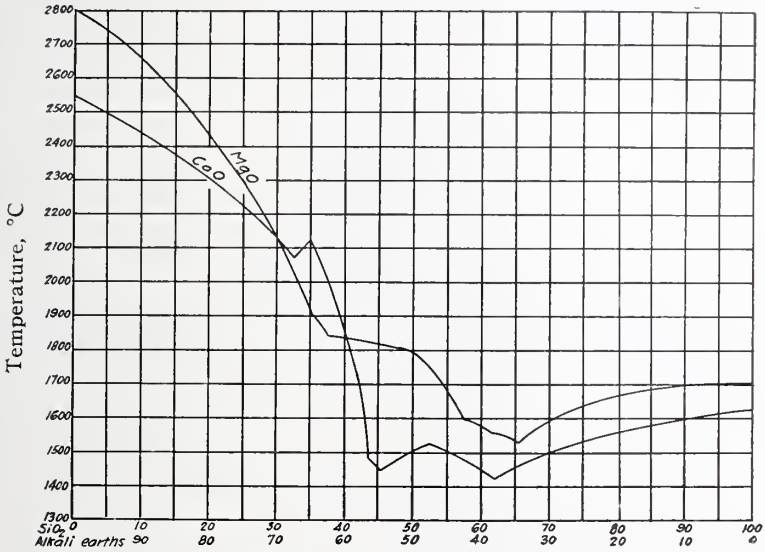

FIG. 2.—The effect of SiO_2 on the fusion of MgO and CaO.

crushing the dolomite and heating it to 950° F. This temperature decomposes the $MgCO_3$ entirely but allows the $CaCO_3$ to remain in the crystalline form. The MgO is then removed with water or by means of a pneumatic separator; another method[3]

[1] A. Wasum, "The Effects of Some Furnace Conditions on Basic Refractories Used in Melting Operations." *Trans. Eng. Ceram. Soc.*, 16, 324–326 (1916–17).

[2] Ardon M. Mitchell, U. S. 1,273,110, July 16, 1918, "Process for Obtaining MgO from Dolomite."

[3] J. O. Handy and R. Isham, U. S. 1,270,819, July 2, 1918, "Separation of Lime from Dolomite."

is to decompose the $CaCO_3$ as well as the $MgCO_3$. Water is then run over the calcined mixture and, owing to its greater solubility, the lime is carried off first. This process is continued until all but 3–10 per cent CaO has been removed.

The effect of Al_2O_3 and SiO_2 on the softening points of MgO and CaO was studied by Rankin and Merwin.[1] It was found (see Figs. 1 and 2) that SiO_2 had a much greater fluxing action on the oxides of the alkali earths than Al_2O_3.

Following are the analyses of five typical commercial refractories prepared from magnesite or dolomite:[2]

	No. 1. %	No. 2. %	No. 3. %	No. 4. %	No. 5. %
CaO	2.60	49.10	0.45	6.00	15.0
MgO	89.20	34.36	87.35	75.00	70.0
SiO_2	5.60	7.23	6.60	7.00	5.5
Fe_2O_3 / Al_2O_3	1.40	5.31 / 2.80	4.30	12.00	8.5
Loss on ignition	1.20	1.11	1.30	....	0.5

Outline of Investigation.

The following impurities were added in varying percentages to a dolomite from Cedarville, Ohio, which had previously been screened through an eight-mesh sieve:

1. Coal Tar
2. Flue dust
3. Iron ore
4. Basic open hearth slag
5. Blast-furnace slag
6. Bedford shale
7. Kaolin
8. Roll scale
9. Calcium chloride

The analysis of Cedarville dolomite is given by the Ohio Geological Survey[3] as follows:

[1] G. A. Rankin and H. E. Merwin, "The Ternary System, $MgO.Al_2O_3.SiO_2$." *Am. J. Sci.*, **45** (1918); G. A. Rankin and H. E. Merwin, "The Ternary System $MgO.Al_2O_3.SiO_2$," *J. Am. Chem. Soc.*, **38** (1916).

[2] No. 1. "Dead-burned California Magnesite," U. S. Magnesite Corporation, 39 Cortlandt Street, New York, N. Y.

No. 2. "Dolomite," Kennedy Refractory Co., Union Arcade, Pittsburgh, Pa.

No. 3. "U. S. Magnesite," E. Kaufmann & Co., Pittsburgh, Pa.

No. 4. "Ferro-magnesite," Refractory Magnesite Corp., New York City.

No. 5. "Dead-burned Magnesite," National Sales Co., Cincinnati, Ohio.

[3] E. Orton and S. V. Peppel, "Limestone, Lime and Sand-Lime Brick," Ohio Geol. Sur., Fourth Series, *Bulls.* **4** and **5**, 152–153 (1906).

	Per cent.
$CaCO_3$	53.95
$MgCO_3$	44.62
SiO_2	0.58
Al_2O_3	0.57
	99.72

Briquettes of the various mixtures were molded and calcined at cone 18 in a Hoskins electric furnace. Immediately upon being taken from the furnace they were weighed and after standing in air, in order to test their air-slaking behavior, they were reweighed after the following intervals: 2, 5, 10, 18, 28, 38, 50,

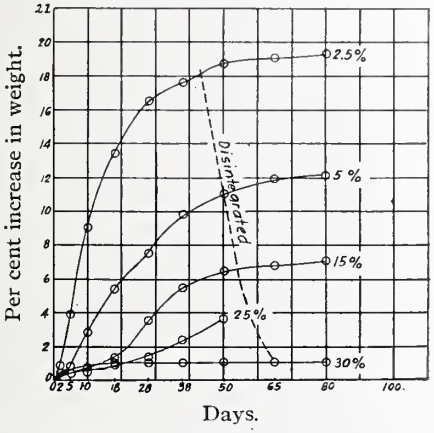

FIG. 3.—The effect of flue dust on the rate of slaking of dolomite, after burning to cone 18.

65, 80, 100 and 125 days, to determine the increase in weight. The point at which the briquettes disintegrated and no longer had mechanical strength was also noted.

In order to determine the effect of size of grain of the raw dolomite on the ease of dead-burning, the dolomite was screened through 4-, 8-, 20- and 40-mesh sieves. Varying percentages of iron ore were added to the dolomite thus screened, and the mixtures calcined to cone 18. The rate of absorption of water and the time necessary for the disintegration of the briquettes was then determined.

The screen analyses of the ground dolomite used were as follows:

Size of grain.	Size of openings in inches.	4 mesh.	8 mesh.	20 mesh.	40 mesh.
4–10	0.25 –0.005	58.80	14.65	...	...
10–20	0.065 –0.0328	18.70	33.82	0.30	...
20–35	0.0328–0.0168	9.50	19.91	34.36	1.96
35–48	0.0168–0.0116	2.25	5.98	12.40	19.50
48–65	0.0116–0.0082	1.95	5.22	12.00	17.10
65–100	0.0082–0.0058	1.88	4.92	8.28	15.80
100–150	0.0053–0.0041	0.71	3.42	4.72	9.10
150–200	0.0041–0.0029	2.24	6.58	11.80	14.25
Through 200	Through 0.0029	3.67	6.04	15.82	22.32

In order to determine the effect of the fluxes on the refractoriness of the dolomite the softening points were also determined.

Results of the Work.

Coal Tar.—A mixture of 10 parts coal tar and 90 parts dolomite was calcined to cone 18 under oxidizing conditions and also

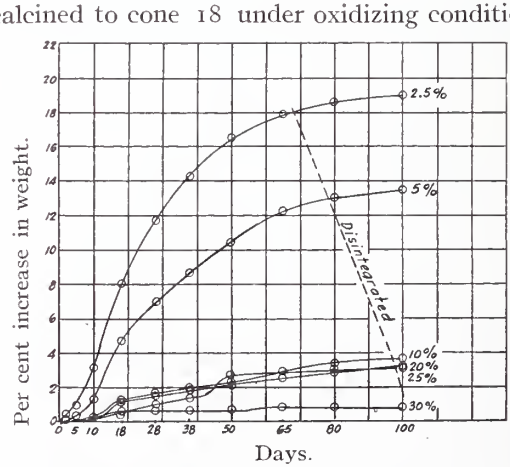

FIG. 4.—The effect of iron ore on the rate of slaking of dolomite after burning to cone 18.

under reducing conditions (secured by packing in graphite). After calcination it was found that the sample which had been calcined under oxidizing conditions disintegrated readily in water

while that which was calcined in graphite did not disintegrate after standing in water for two months. The latter material, however, was very porous and the addition of some flux would be necessary to produce a brick which would be impervious to slag.

Flue Dust.—For the results secured with flue dust, see Figs. 3, 10 and 11. When calcined, the mixture had a dark brown color

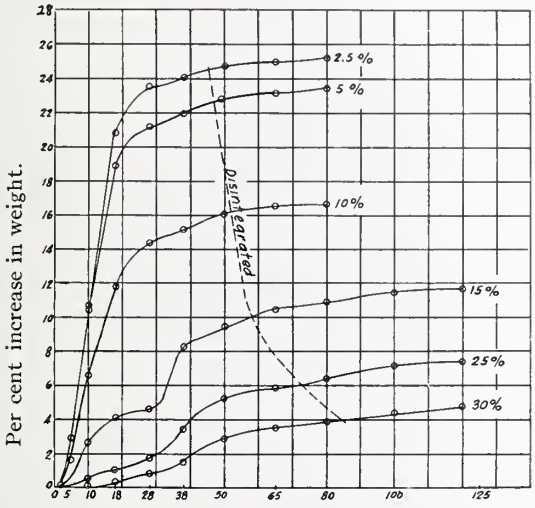

FIG. 5.—The effect of basic open hearth slag on the rate of slaking of dolomite after burning to cone 18.

and, in some cases, showed dusting.[1] This dusting was due to conversion of the beta to the gamma form of calcium orthosilicate, $2CaO.SiO_2$, which is formed at 675° C in cooling and is accompanied by an increase in volume of 10 per cent. This volume change shatters the material into a fine dust and sometimes breaks off big pieces from the sintered dolomite. It would appear that this would be a serious factor in causing spalling of dolomite brick made from a mixture of this kind.

[1] G. A. Rankin, "The Ternary System, $CaO—Al_2O_3—SiO_2$," *Am. Jour., Sci.*, **39**, 7–8 (1915).

The mixture made by calcining the mixture of 30 parts flue dust and 70 parts dolomite disintegrated after standing in air for 65 days. This mixture had a softening point of cone 31+.

Iron Ore.—For the results secured with iron ore, see Figs. 4, 10 and 11. The briquettes when burned had a brown color and showed very little dusting. It is evident that free silica, when added to dolomite in amounts capable of forming the orthosilicate, causes dusting, so it appears that the silica was held in

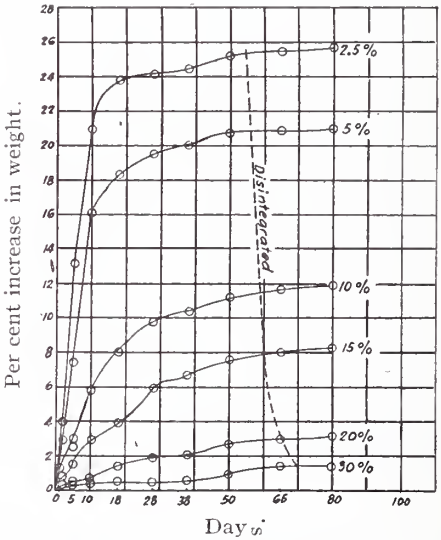

FIG. 6.—The effect of blast furnace slag on the rate of slaking of dolomite after burning to cone 18.

combination with the iron in this case and very little dusting resulted.

Mixtures made by calcining 30 parts of iron ore with 70 parts of dolomite had not disintegrated at the end of 100 days and had a softening point of cone 31−.

Basic Open Hearth Slag.—In Figs. 5, 10 and 11 are given the results secured with basic open hearth slag. The briquettes had a black color and showed some dusting. The mixture made by calcining 30 parts of slag with 70 parts of dolomite had a softening point of cone 31+ and disintegrated at the end of 85 days.

Blast Furnace Slag.—The results secured with blast furnace slag are shown in Figs. 6, 10 and 11. The calcined briquettes had a white color and dusted excessively. This was undoubtedly due to the high silica content which was conducive to the orthosilicate formation in this slag.

The mixture made by calcining 30 parts of blast-furnace slag

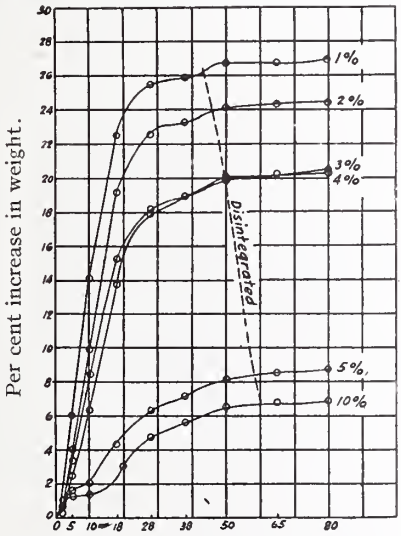

FIG. 7.—The effect of shale on the rate of slaking of dolomite after burning to cone 18.

with 70 parts of dolomite disintegrated in a comparatively short time, 70 days.

Bedford Shale.—The calcined briquettes containing shale and dolomite had a buff color and showed excessive dusting. This was also due to the high silica content of the shale—forming the orthosilicate with lime. Those briquettes containing the highest per cent of shale showed the most dusting.

The mixture prepared by calcining 10 parts of shale with 90 parts dolomite disintegrated at the end of 60 days (see Figs. 7, 10 and 11). The fluxing effect of silicious materials, like shale, seemed to decrease the refractoriness to a greater extent than the

addition of iron oxide—the mixture prepared by calcining 10 parts shale with 90 parts dolomite having a softening point of cone 31+.

Georgia Kaolin.—The calcined briquettes containing kaolin and dolomite had a cream color and showed less dusting than the mixture containing shale. The body burned dense and hard and if used for brick would be impervious to slag penetration. The mixture prepared by calcining 10 parts kaolin with 90 parts

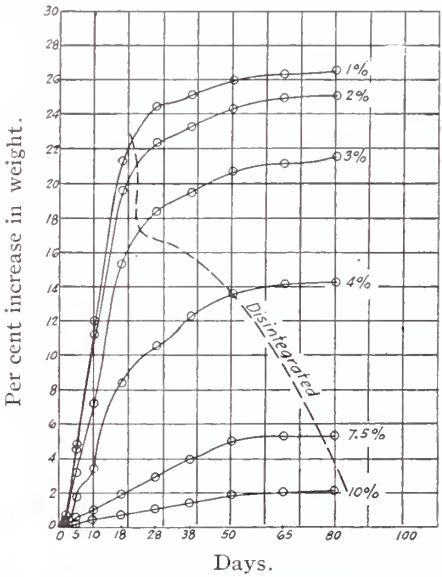

FIG. 8.—The effect of kaolin on the rate of slaking of dolomite after burning to cone 18.

dolomite disintegrated at the end of 85 days (see Figs. 8, 10 and 11). This mixture had a softening point of cone 31. When less than 3 per cent kaolin was used, the briquettes disintegrated in a comparatively short time, but where more was used there was a large increase in this time as is shown by the abrupt break in the disintegration curve.

Roll Scale.—For results secured with roll scale see Figs. 9, 10 and 11. The calcined briquettes in this case had a brown to

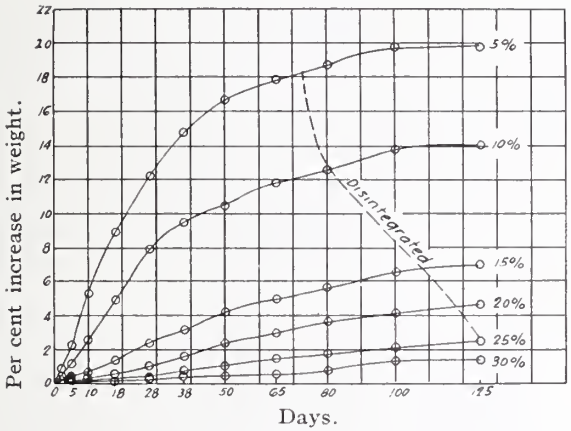

FIG. 9.—The effect of roll scale on the rate of slaking of dolomite after burning to cone 18.

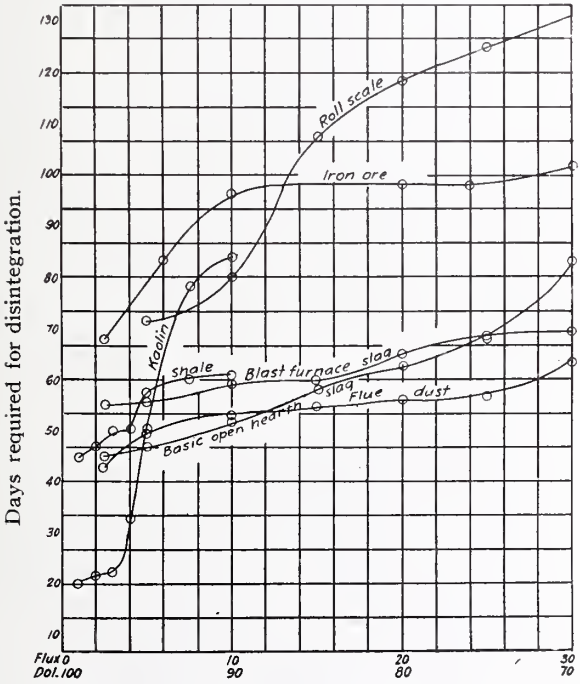

FIG. 10.—The effect of impurities on the time of disintegration of dolomite after burning to cone 18.

black color and very little dusting was noted—this being due to the high iron content of the roll scale. The mixture prepared by calcining 30 parts roll scale with 70 parts dolomite was not disintegrated at the end of 125 days—this mixture standing up better than any of the others (see Fig. 7).

Calcium Chloride.—A mixture of 10 parts calcium chloride and 90 parts dolomite was calcined to cone 14. The product had a buff color, good mechanical strength, and was very resistant

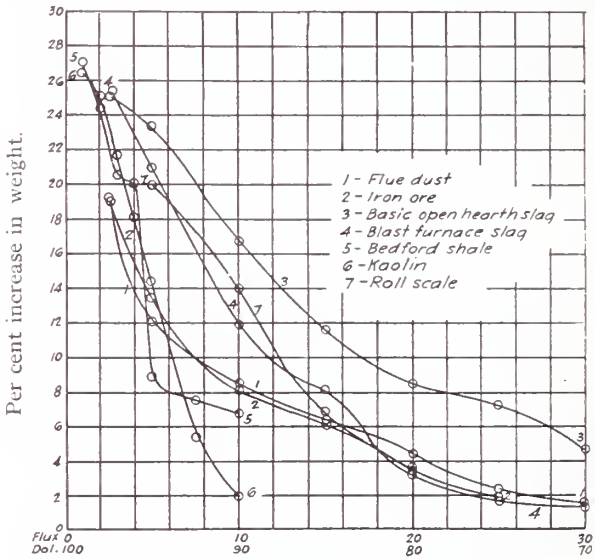

FIG. 11.—The effect of impurities on the increase in weight in air of dolomite after burning to cone 18.

to slaking. It, however, was very porous and if used without a flux would have low resistance to the penetration of slag.

Effect of Size of Grain on the Ease of Dead-Burning Dolomite.

The effect of size of grain on the ease of dead-burning was studied by screening the dolomite through 4-, 8-, 20- and 40-mesh sieves before calcination. For the results see Fig. 12. It appears that the coarse material passed through 4- and 8-mesh stood up longer than the dolomite screened through a 20-mesh

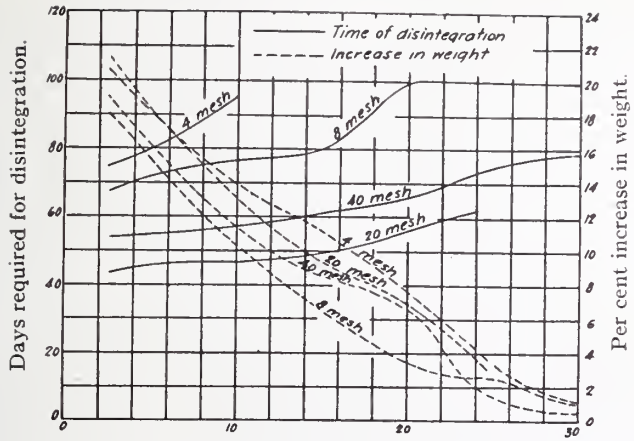

FIG. 12.—The effect of size of grain on the time of disintegration and increase in weight of dolomite in air after burning to cone 22.

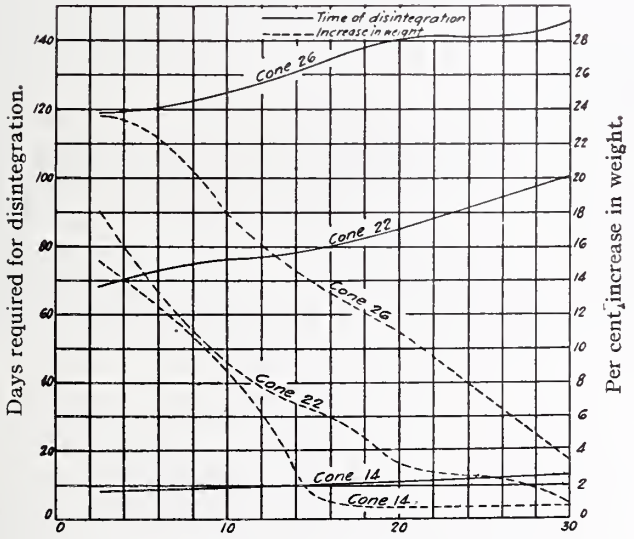

FIG. 13.—The effect of burning temperature on the time of disintegration and increase in weight in air of dolomite after calcination.

sieve, while the fine dolomite, screened through a 40-mesh sieve, stood up better than that screened through a 20-mesh sieve.

It appears, therefore, that the best results are obtained by grinding dolomite extremely coarse, through 4-mesh, or fine, through a 40-mesh sieve. In the first case a coating is probably formed about the individual grains. This coating protects the centers of the grains which are high in the purer CaO. Where no further fine grinding is necessary, as is the case when granular dolomite is used for basic linings, it would seem that this material would be satisfactory and have the advantage over the finely-ground dolomite in that the cost of grinding would be less. On the other hand, if this material were finely ground and mixed with water, as is done in making brick, it would crack in drying—owing to the slaking of the uncombined lime. For such purposes finely grinding the dolomite with iron oxide before calcination would be preferable because the dead-burning would be uniform throughout the material.

It is claimed by the users that domestic magnesite, which is very pure, lacks bond when used for furnace linings and soon crumbles. It would seem that the coarse grains of dolomite, coated with iron oxide, would stay set in the furnace—owing to the tendency of the grains to stick together.

Effect of Temperature of Calcination on the Ease of Dead-Burning Dolomite.

The effect of temperature of calcining on the ease of dead-burning was studied by burning to cones 14, 22 and 26. In every case the increased temperature of burning increased the number of days necessary for disintegration and decreased the maximum absorption of the briquettes, (see Fig. 13).

Summary.

Roll scale seems to be very effective in dead-burning dolomite without decreasing its refractoriness too much.

It was found that the addition of impurities containing silica caused dusting in the calcined material—due to conversion of the beta $2CaO.SiO_2$ to gamma $2CaO.SiO_2$. This was most pronounced when blast-furnace slag or shale were used and was

noticed to a lesser extent when flue dust, kaolin or basic open hearth slag were used. Mixtures containing roll scale or iron ore showed the least dusting.

It was found that a large percentage of iron oxide (20–30 per cent with 80–70 per cent dolomite) may be used without decreasing the refractoriness of the material below cone 31. Impurities which are silicious in character, like kaolin, decrease the refractoriness to a much greater extent. For instance, a mixture made by calcining 10 parts kaolin with 90 parts dolomite had a softening point of cone 31.

When dolomite is mixed with tar, packed in graphite, and burned to cone 18, it becomes very resistant to slaking. If it is not packed in graphite during the burning it slakes easily.

When dolomite is mixed with 10 parts calcium chloride and burned to cone 15 it becomes very resistant to the slaking action of water.

It was found that dolomite ground extremely coarse (through 4- or 8-mesh), or fine (through 40-mesh), was easier to dead-burn than when screened through 20-mesh. The coarse material, however, if reground, does not resist slaking as well as the 40-mesh material.

Increasing the burning temperature increases the resistance of the dolomite mixtures to disintegration, as would be expected.

In conclusion, the author wishes to acknowledge his indebtedness to Mr. R. T. Stull for his assistance in the above work.

Bureau of Mines,
Mining Experiment Station,
Columbus, Ohio.

THE COMPARATIVE VALUE OF KNEADING AND PUGGING IN THE PREPARATION OF PORCELAIN BODIES.

By L. E. Barringer and Chester Treischel.

Introduction.

Proper preparation of pottery mixtures is an important step in the process where the highest degree of excellence and uniformity is required. The several ingredients must not only be thoroughly mixed but the resultant body must be of uniform consistency in order to produce uniformity from piece to piece and should be free from cracks, air-pockets, and laminations which all produce imperfections in the finished ware.

Blunging and agitating mix the ingredients of pottery bodies but there is a tendency toward segregation in the filter-pressing, the lighter particles passing to that part of the filter-press cake farthest from the slip inlet and the heavier particles tending to concentrate nearest the feed hole.

In reducing filter-press cakes to the proper consistency for forming the ware, it is desirable to use an apparatus which both kneads and mixes and at the same time leaves the mass comparatively free from imperfections or air-inclusions. For reducing filter-press cakes to the desired consistency, two machines have been in use for many years, namely, the pug mill and the kneading- or rolling table.

The pug mill is an excellent mixer and works rapidly but tends to leave laminations where the knives and augers pass through the putty-like mass. Pug mill designers have studied this question for years and have succeeded in considerably reducing the amount of lamination by modifying the design of the augers; by special lubrication of the die or nozzle; by changing the shape of the pug mill barrel, etc., but still the tendency toward lamination remains as an inherent fault in this type of machine.

The rolling or kneading table has been used abroad for some years and has recently been described in connection with the

preparation of electrical porcelain bodies in France,[1] but has not been used to any considerable extent in this country—more because of its limited capacity than because of its not having any particular merit.

Theoretically, such a type of machine would not be expected to

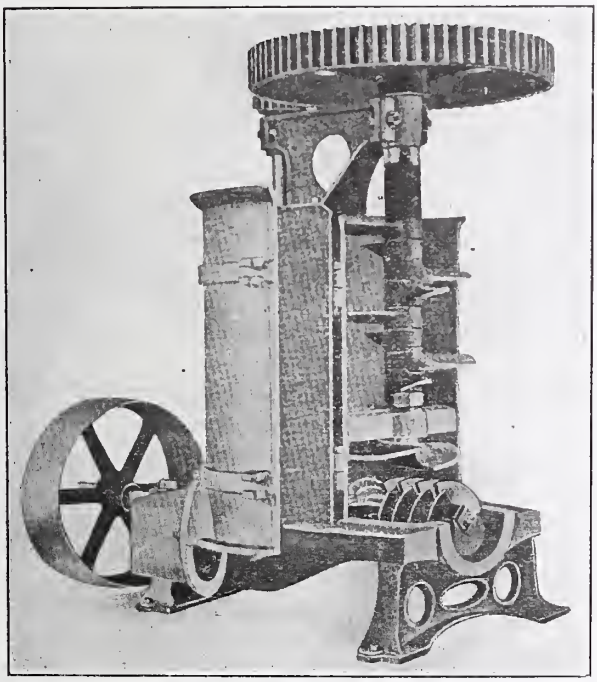

FIG. 1.—Pugging machine.

mix as well as a pug mill but would be expected to leave the mass more free from imperfections, both through not itself introducing such imperfections and also by handling the material in such a way as to expel more thoroughly any air which might be present in the mass—as the result of simply batting down filter-press cakes of irregular surfaces.

[1] Revue Generale de l'Electricite," 1, No. 2, p. 60.

Experimental.

Desiring to obtain first-hand knowledge of the comparative values of the pug mill and kneading table, particularly for preparing electrical porcelain bodies, the authors, together with Mr.

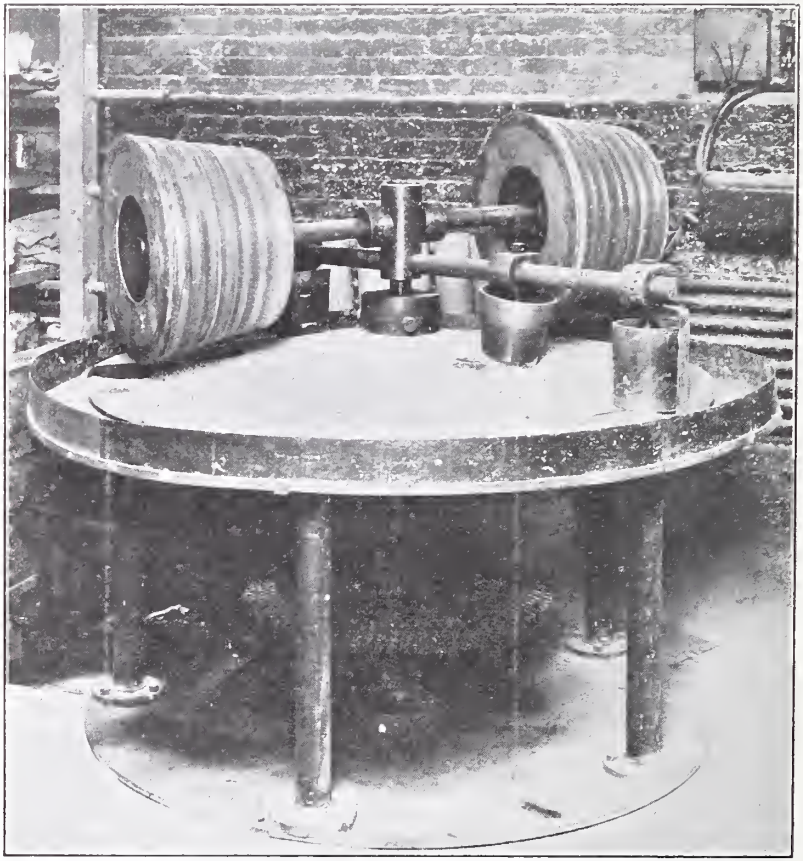

FIG. 2.—Kneading Machine.

F. Cermak, of the General Electric Company, set about trying both machines in the Porcelain Department of the General Electric Company at Schenectady, N. Y.

The type of pug mill used is shown in Fig. 1 and represents the machine used regularly in the preparation of porcelain bodies.

The kneading table used is shown in Fig. 2 and is one constructed by the General Electric Company to duplicate a French machine of the type commonly employed in European potteries.

Since the air content of the product of each machine can be taken as an indication of the condition inside the blanks, it was decided to use Spurrier's method[1] for obtaining the amount of entrapped air. Small samples about 30.0 cc. in volume were taken from the edges and from the centers of all blanks. These blanks, in turn, were taken from hard, medium, and soft bodies—the designation in each case referring to the workable condition of the body.

The data presented in Table 1 are averages of a large number of determinations—the actual testing covering a period of two months.

TABLE 1.

Machine.	Consistency of body.	Sample.	Average % air (by volume).
Pug mill No. 1		Interior	29.8
Pug mill No. 1		Edge	29.6
Pug mill No. 2		Interior	31.1
Pug mill No. 2		Edge	29.2
Kneading Table	Hard body—20 min.	Interior	25.6
Kneading Table	Hard body—20 min.	Edge	25.9
Kneading Table	Medium body—20 min.	Interior	27.5
Kneading Table	Medium body—20 min.	Edge	27.8
Kneading Table	Soft body—20 min.	Interior	28.3
Kneading Table	Soft body—20 min.	Edge	30.6
Kneading Table	Hard body—40 min.	Interior	29.5
Kneading Table	Hard body—40 min.	Edge	28.1
Kneading Table	Medium body—40 min.	Interior	27.4
Kneading Table	Medium body—40 min.	Edge	27.0
Kneading Table	Soft body—40 min.	Interior	32.1
Kneading Table	Soft body—40 min.	Edge	31.0

[1] *J. Am. Ceram. Soc.*, 1, 710 (1918).

Conclusions.

The results of these tests together with the conclusions which have been drawn are:

1. The center of the pug-mill blank contains more air than the edge, this being due to the greater pressure on the blank where it comes in contact with the die.

The center of the kneading-table blank may or may not contain *less* air than the edge, according to the length of time of kneading and to the amount of water contained in the body which is being tempered—a hard body contains less water than a soft one. Hard or medium bodies on 20-minute runs and medium bodies on 40-minute runs gave the most consistent results.

2. The kneading table is a more efficient air-remover than the pug mill. This is evidenced by a comparison of the data on pug mill body No. 1 and kneading table, hard body, 20-minute run—the bodies in this case being identical. However, a continued kneading for 40 minutes does not produce the desired effect, since air seems to be re-incorporated in the blank. This will be evident from a comparison of pug mill body No. 1 and the kneading table, hard bodies, 20 and 40-minute runs.

3. The kneading table seems to be most efficient on medium and hard bodies and when run for a period of 20 minutes. An average of the specific gravity and porosity determinations of all runs on the kneading table compared with the average for the pug mills shows a higher specific gravity and a lower porosity for the former.

General Electric Co.,
Schenectady, N. Y.

COMMUNICATED DISCUSSIONS.

C. F. Binns: Some years ago in England I had experience with the rolling table in the working of bone china. The pug mill as then designed was not suitable. It made the clay shorter instead of more plastic and bone china is a short body anyway. The only effective method before the rolling table made its appearance was hand-wedging, but the use of the table banished this entirely. It is quite true that for a large output the rolling table is slow but this is chiefly on account of the fact that the operation is not continuous. The table must be stopped, emptied and refilled for every charge. For bodies of the more plastic type no

doubt the pug mill is sufficient but the improvement of all wares lies in the direction of the use of less plastic clays and a higher percentage of ground material. Furthermore, these finer bodies are not likely to be used in such large quantities as are those of the commoner wares so that it does not seem unreasonable to expect that the rolling table will find profitable use for the finer products.

Alfred University,
Alfred, N. Y.

B. E. Salisbury: Something over ten years ago experiments were made in our plant with a kneading machine, which, I understand, was procured from abroad, and these seemed to indicate possibilities of attaining a more compact and plastic body. The experiments were not carried far enough, however, to definitely determine the possibilities of its use. The output, of course, as stated in the paper, is only a fraction of that of a pug mill, and therefore the labor cost of preparation of clay would be considerably higher.

We did not conclusively demonstrate that the better results from the kneading machine would offset the extra cost of the preparation of the clay by its use. I believe, however, that there are some possibilities along this line and am glad to have the matter come up for discussion.

Onondaga Pottery Co.,
Syracuse, New York.

C. E. Jackson: First, let me say that I am not in a position to discuss this question from a scientific angle for the reason that I do not have comparative data available. I have had four or five years of personal experience with an English or French kneading machine which was similar in design to the one discussed in the paper. We made a direct comparison between this machine and an American circular-barrel pug mill. The thing that militates against a complete installation of kneading machines in place of the pug mills is the fact that the kneading machines have only about one-sixth the capacity of the ordinary pug mill and very few factories are equipped to give sufficient space to machines of this character—especially to the number that would be required for ordinary production. In other words, the

relation between the comparative output of the pug mill and kneading machine is that the pug mill has about six times the output; as to the quality of clay delivered, there is no comparison. I believe that the clay after it has been on the kneading machine is better in all respects than clay that is hand wedged. The kneading machine also strengthens a weak body and produces a clay which works more smoothly.

Furthermore, for the manufacture of difficult pieces, the clay prepared by the kneading machine lends itself much more readily to their production than clay from the pug mill. For extremely delicate work, in which quality is the sole consideration, I believe the kneading machine to be the thing; for the average commercial work, where the requirements are not so exacting, I believe that the pug mill can be used to advantage for the reasons above noted.

WARWICK CHINA COMPANY,
WHEELING, W. VA.

THE DEVELOPMENT OF IMPROVED GOLD RUBY GLASS.

By H. T. Bellamy.

It has been a well-known phenomenon for sometime past that gold and copper produce a ruby color in glass which contains certain necessary constituents and which has been given a special heat treatment. The color is universally agreed to be due to the metals in suspension as colloids.

The ability of gold to exist in the colloidal state was first observed by Faraday while experimenting on the volatilization of gold. He found that gold in an atmosphere of hydrogen volatilized quite readily and that the vapor condensed as an intense ruby stain. Mostowitsch and Pletneff[1] have recently reported similar results. They melted gold in an unglazed porcelain boat, in streams of air, carbon dioxide, carbon monoxide, nitrogen and oxygen and held it at various temperatures up to 1400° C for a period of thirty minutes. No loss of gold could be detected. However, when heated in hydrogen the gold began to volatilize at 1250° C and the volatilization increased with temperature and time. The unglazed boat and the heating tube were colored an intense red—due to colloidal gold. It is probable that Au_2H_2—which is unstable, breaking up into Au_2 and H_2—was first formed. By heating copper in hydrogen a similar action was observed.

As an example of colloidal phenomena, gold ruby glass has been rather thoroughly investigated and the behavior of colloidal gold in this solid solution appears to be similar to its behavior in aqueous solutions, existing in both instances as colorless, green, red, blue, violet, or brown particles. When ruby glass is at the melting temperature the particles are colorless and when suddenly chilled they remain colorless, but if the glass is re-heated to the softening point, the particles become ruby. Further heating tends to produce other colors.

[1] *J. Russ. Metall. Soc.*, **1915.**

Zsigmondy[1] says that at the melting temperature the gold is all in solution but at ordinary temperatures the colorless glass contains gold in two different forms—most of it being present in the supersaturated crystalloid solution and part existing as nuclei, which at higher temperatures serve as centers of growth.

Experimental.

The work which supplied the information for this paper was not undertaken to study ruby glass in general nor as a purely research proposition and on this account an exhaustive exposition of the problem is not to be expected. The manufacture of ruby glass was taken up to reproduce a ruby glass purchased in France, the importation of which had been discontinued since the war. A glass of the exact shade and possessing the requisite working properties could not be obtained from domestic suppliers. The treatment, to which the glass is subjected, is rather severe, as it must not shatter when introduced into a glass blower's flame, it must not discolor when heated to a very soft condition and it should be of such a nature that small lenses can be formed from the heated rod without subsequent annealing. The lenses so formed should be of uniform color, matching an accepted standard; should be free from surface cracks and other visible defects; and when mounted in a brass shell should withstand impacts of three pounds applied at the rate of 115 per minute.

A chemical analysis was made of the imported ruby glass and the molecular formula calculated as follows:

Analysis:

	Per cent.
SiO_2	38.35
PbO	54.48
K_2O	6.17
Sb_2O_3	0.67
R_2O	0.70
Gold	Present
B_2O_3	Not determined

Calculated Formula:

$$\left.\begin{matrix} 0.791\ PbO \\ 0.209\ K_2O \end{matrix}\right\} \quad 0.0173\ R_2O_3 \quad \left\{\begin{matrix} 2.05\ SiO \\ 0.0075\ Sb_2O_3 \end{matrix}\right.$$

[1] Zsigmondy, "Colloids and the Ultra-microscope."

While this work was being done the scarcity of potash was beginning to be evidenced and although the imported glass was made with a potassium compound, as was to be expected, it was decided to try to substitute a sodium compound.

The first melts were made in a small crucible-type gas furnace, using Battersea crucibles. After making a few trial batches to determine the proper quantity of gold to be added, a glass of the following formula was developed:

Ruby No. 1.

$$\left.\begin{matrix} 0.583\ PbO \\ 0.417\ Na_2O \end{matrix}\right\} \quad 0.0038\ B_2O_3 \quad \left\{\begin{matrix} 1.95\ SiO_2 \\ 0.0074\ Sb_2O_3 \end{matrix}\right.$$

The gold, which is not included in the formula, was prepared by dissolving ten karat sheet gold in aqua regia, separating the base melts in the ordinary manner, and diluting the gold chloride so that ten cubic centimeters contained one gram of metallic gold. The requisite volume of gold chloride was added to the sand of the batch and evaporated to dryness.

Ruby No. 1 appeared to match the imported ruby in color and preparations were made to melt a larger quantity. A No. 30 open glass-melting pot was used in the same type furnace and a batch eight times as large as the preceding one was prepared. The batch was added gradually and held at approximately 1300° C for fifteen hours when it was ready to work. Rods were made by the ordinary method of forming a bell-shaped gather. Each gathering of glass was allowed to solidify and, as the next coating was added, the previous coating developed the ruby color. After completion, the gather was re-heated in an improvised glory hole and drawn into rods.

It was very difficult to heat the gather sufficiently uniformly so that the rod would be of one color throughout its length. The smaller area next to the punty was usually overheated—showing light blue, purple and rose colors. Furthermore, the consecutive layers of the gather were frequently of different colors and when a lens was formed from the rod and viewed over a light it appeared streaked. The center of the rod showed the greatest tendency to discolor, as was to be expected—since the small initial gather

could readily become over-heated when dipped into the molten metal. However, when proper precaution was exercised in having the metal at a certain temperature, when the gathers were cooled to the proper degree, and when the re-heating was carefully controlled, a certain percentage of rod matching the standard shade was obtained from each gather.

Simultaneously with the development of ruby glass, experimental work was being carried on to produce other colored glasses to match imported glasses not obtainable since the war and also to develop opalescent colored glass of certain requirements which we were unable to procure from either domestic or foreign suppliers. The opalescence required was to be of such a degree that a lens should be of uniform color and opalescence when viewed normally over a light and the diffusion should be such that the quantity of light transmitted normally would compare favorably with that transmitted at 90 degrees from normal. To compare opalescent colored glasses on this basis, the sample lens was set in a tube in front of a small lamp mounted in a suitable revolving support in a photometer.

With the above requirements in mind, the development of an opalescent red was begun and after melting a few small batches to adjust the color and opalescence a glass having the following formula was tried out:

Opalescent Red No. 1.

0.502 PbO 0.342 Na_2O 0.156 CaO	0.0154 Al_2O_3	1.46 SiO_2 0.0134 Sb_2O_3

Lenses of Opalescent Red No. 1 had the proper color and degree of opalescence but the glass rods shattered while being heated in the glass blower's flame and the finished lenses shattered when subjected to the previously described impact test.

Although the quantity of cryolite used as an opacifier in opalescent red was considerably less than is present in our opalescent white glass, which withstood the impact test extremely well, the brittleness was attributed to its presence and it was decided to substitute tin oxide. After a few trial batches a glass having the following formula was developed:

Opalescent Red No. 2.

$$\left.\begin{matrix} 0.668\ PbO \\ 0.332\ Na_2O \end{matrix}\right\} \quad 0.0028\ B_2O_3 \quad \left\{\begin{matrix} 2.17\ SiO_2 \\ 0.0831\ SnO_2 \end{matrix}\right.$$

Opalescent Red No. 2 melted well, was readily worked into rods, and lenses formed from the softened rods fulfilled the requirements previously described. The resistance to impact was equal to that of the transparent ruby and the light diffusion was all that was desired. The opalescent red, being lighter in shade, is not as pleasing to the eye as the so-called ruby.

In developing Opalescent Red No. 2, a much smaller amount of gold was used than is present in Ruby No. 1, but as the concentration of tin oxide was increased to produce opalescence, a dark red glass resulted which in the form of a lens was almost opaque. The gold content was repeatedly reduced until Opalescent Red No. 2 of the desired properties were obtained. However, at an intermediate stage, when the gold content was about one-fifth of that used in the transparent Ruby No. 1, a glass was obtained which was quite similar in shade to Ruby No. 1. A perfect match for this glass and the former standard, the imported ruby, was produced by a slight adjustment arriving at the following molecular formula:

Ruby No. 2.

$$\left.\begin{matrix} 0.536\ PbO \\ 0.464\ Na_2O \end{matrix}\right\} \quad 0.029\ B_2O_3 \quad \left\{\begin{matrix} 1.96\ SiO_2 \\ 0.107\ SnO_2 \end{matrix}\right.$$

Lenses molded from Ruby No. 2 did not crack or shatter when subjected to the standard impact test and, unlike the lenses made from Ruby No. 1, were never streaked or off-color. The melting was carried on in No. 30 open glass-pots in a crucible-type gas furnace for a period averaging fifteen hours. The gather for rod drawing was shaped in the usual manner and each successive coating was allowed to cool before the next layer was added. The ruby color developed in the previous gatherings, as was the case when working Ruby No. 1, but always to a uniform color. The gather was re-heated to plasticity and drawn into rods in the usual manner. The rods and lenses made from Ruby No. 2 were always of a uniform ruby color. At first the precautions required in working Ruby No. 1 were followed but it was soon evident that

Ruby No. 2 would always develop the ruby color on re-heating and that the temperature of the glory-hole would not alter this color.

Ruby No. 3.

Quite recently we were called upon to produce a red glass enamel and finely powdered Ruby No. 2 was used with other suitable constituents. The enamel was not sufficiently red for the small thickness which was viewed over a light, and the amount of gold was trebled. The result was a very dark ruby referred to as Ruby No. 3 and which worked exactly like Ruby No. 2. The color developed upon heating to the softening point and it could not be changed except at a very high temperature when the ruby color disappeared, leaving a colorless glass.

The presence of a certain percentage of tin oxide, besides acting in the ordinary manner as an opacifier, apparently reacts to precipitate the gold in the red colloidal state. Its presence may affect the magnitude of the gold particles catalytically and produce this result or it may react to precipitate the gold particles as red hydrosols in the same manner that certain substances aid or retard the precipitation of colloidal gold in aqueous solutions.

Walker[1] states that gold particles may exist so finely divided in solutions that the ultramicroscope not only fails to reveal their presence as individuals, but fails even to detect any scattered luminosity in the solution when a powerful beam of light is concentrated in the solution and, since luminosity can be detected with gold particles of two millionth's of a millimeter in diameter, it is concluded that red colloidal gold particles have dimensions approximating the recognized magnitude of molecules, or approximately one millionth of a millimeter.

Ruby No. 2 and Ruby No. 3 and the opalescent red glasses which we have produced can exist in only two states—the red and the colorless. When at a high temperature, the absence of color is probably due to the fact that the gold is in solution. When colorless at ordinary temperatures, the gold particles, some of which it is assumed have separated out, are too small to be visible. Upon reheating, the gold particles grow until of visible size

[1] Walker's, "Physical Chemistry."

in which form they impart a ruby color to the glass. If the ruby colored glass is heated to the temperature at which the glass regains its solvent action, the red gold hydrosol again goes into solution. These glasses appear to be so constituted that the gold cannot assume the various colors which was noted in Ruby No. 1 and which it is generally believed is to be expected in gold ruby glass.

Conclusions.

It is rather difficult to draw general conclusions from this work regarding the composition of workable ruby glass for, as previously mentioned, this study was undertaken solely to produce glasses with predetermined properties. However, it may be stated that the range of stable red glasses is rather wide as the red opalescent described is quite light and Ruby No. 3 is very dark. The quantity of gold used to produce these shades of red varied from one part in ten thousand to one part in three thousand. The use of tin oxide to stabilize the gold and produce a ruby color with the minimum quantity of gold is new and novel and a patent has been granted. Two definite conclusions are apparent, namely: (1) a stable, reliable, gold ruby glass can be economically produced and (2) the presence of other colors is not a natural phenomenon of gold ruby glass, for when the necessary components are properly proportioned, the gold particles are either red or colorless.

CHEMICAL DIVISION—HAWTHORNE WORKS,
WESTERN ELECTRIC COMPANY, INC.,
CHICAGO, ILL.

COMMUNICATED DISCUSSIONS.

F. CARDER: The paper is very interesting and shows that the author has given the subject careful thought.

The very small lenses which were shown by Mr. Bellamy should not have been difficult to make or match, either for color or for the physical standards set, especially when so high a content of PbO could be used in the batch.

Has Mr. Bellamy tried his Ruby No. 1 after gathering a mass from the pot, and cooling until it was below a red heat and then raising the temperature slowly by heating in a glory-hole? Has

he subjected his various trials to blowing out with the blowpipe or flashing his ruby on a flint base? While the rods or lenses may be good enough in color for the purpose intended, I am inclined to think that upon treating his ruby as suggested, he would find discolorations such as he mentions, *viz.*, blue, purple or brownish streaks which usually occur when gold ruby is subjected to a too sudden transition from the colorless state (or state of solution) to the colloidal or crystalline state.

The proportion of gold to the batch, 1 to 3000, or even 10,000, is, to my mind, excessive. Josiah Wedgewood found that one part gold to 20,000 batch colored glass red.

I was somewhat surprised to note that Mr. Bellamy considers the introduction or use of SnO_2 in gold ruby as an innovation. When gold ruby was discovered it was made with "purple of cassius," which some chemists believe to be a gold stannate. I believe that Kunkel made his first gold ruby with "purple of cassius," samples of which are still to be seen in the art museums in Dresden. Berzelius states that "purple of cassius" contains 28.2 per cent of gold to 64 per cent of tin oxide. To my knowledge tin oxide has been used for a great many years and in my experience in manufacturing glass I have never known any formula worthy the name of gold ruby which did not contain tin oxide. Many other oxides are used to give reds varying from a yellowish red to a purple, such as U_2O_3, Fe_2O_3, and CoO.

Steuben Glass Works,
Corning, N. Y.

E. C. Sullivan: Mr. Bellamy's method of intensifying the red color of gold ruby by means of tin oxide recalls the similar use of tin oxide with copper in making qualitative tests by means of the blowpipe applied to the bead of molten borax glass. In testing for copper, tin oxide is added to the bead to assist in the formation of the color of reduced copper, and a test for tin depends upon subjecting the borax bead containing copper to such a reducing condition that the ruby color of the copper will develop if tin is present, not if it is absent.

Corning Glass Works,
Corning, N. Y.

R. L. FRINK: It occurs to me that the author's statement in the last paragraph, last three lines: "The presence of other colors is not a natural phenomenon of gold ruby glass, for when the necessary components are properly proportioned, the gold particles are either red or colorless," is not compatible with the results as found by Zsigmondy and others; that gold, when existing in colloidal solution, or in aqueous solution, irrespective of the components, may produce colors ranging from red to blue.

I also cannot understand how it is possible to obtain patents upon the use of tin, or its salts, to either stabilize or in any manner affect the characteristics of gold in glass, for I had supposed that this was an old, in fact very old, method of producing ruby glass, at least I have heard of it for the past 25 years.

THE FRINK LABORATORIES,
LANCASTER, OHIO.

H. T. BELLAMY: 1. Practically all gold ruby glass described in the literature contains potash as an alkali. In view of the scarcity of potash at the time, soda was substituted, and the resulting glass was equally as satisfactory as ruby glass made with potash.

2. A ruby glass designated as "Ruby No. 1," which matched the imported ruby glass, and like it and other ruby glasses described in the literature, was changed to blue, purple or brown under certain conditions, was developed.

3. An improved "Ruby No. 2" was developed which does not discolor. This result is ascribed to the high percentage of tin oxide. Of course, those familiar with the art, know that "purple of cassius" has been extensively used in making ruby glass and that it contains a certain amount of tin oxide but experience has shown that the ruby glass so made is not stable. The author claims that when sufficient tin oxide is present in a properly proportioned batch, the ruby glass will not discolor, and that this feature is an innovation.

4. Another advantage in using a high percentage of tin oxide is the production of ruby glass with a minimum quantity of gold. While there is no doubt that red glass has been made with one part gold in twenty thousand parts batch, the literature gives

ruby glass formulas ranging from one part gold in one thousand parts batch to one part gold in fifteen hundred parts batch. The usual quantity was being used and the introduction of tin oxide reduced the gold content to one-fifth of its original amount.

It should be borne in mind that this paper is the story of a manufacturing investigation and that the statements made were based on information obtained from a large number of glass batches. The title "Gold Ruby Glass" under which the paper was presented, is no doubt rather broad and it has been revised to "The Development of Improved Gold Ruby Glass." It might appear that the glass described should be restricted to the manufacture of glass rod and small lenses. However, Ruby No. 2 has been successfully made into tubing as well as blown, flashed and pressed parts, and in conclusion the author desires to emphasize that this glass has been subjected to all conceivable temperature transitions and never has a particle of glass been observed which was other than ruby or colorless.

AMERICAN CERAMIC SOCIETY.

Acquisition of New Members during April, 1919.

Resident Associate.

Allison, LeRoy W., "Brick and Clay Record," 170 Roseville Ave., Newark, N. J.

Bausch, Frederick E., 1105 Chemical Bldg., St. Louis, Mo.

Dolman, C. D., Northwest Magnesite Co., Chewelah, Wash.

Geller, Roman F., Bureau of Standards, Pittsburgh, Pa.

Graham, E. H., Washington Iron Works, Los Angeles, Cal.

Gregori, L. P., Midland Terra Cotta Co., Chicago, Ill.

Harding, Browne, Providence Base Works, Providence, R. I.

Huston, C. H., Chattanooga Stamping & Enameling Co., Chattanooga, Tenn.

James, W. Roy, 118 W. 14th St., Marion, Ind.

Jensen, James L., Empire China Works, Brooklyn, N. Y.

Owens, Frank W., Canton Stamping & Enameling Co., Canton, Ohio.

Rand, C. C., National Lamp Works, Cleveland, Ohio.

Risch, Edward J., Midland Terra Cotta Co., Chicago Ill.

Sharp, Chester L., The Frink Laboratories, Lancaster, Ohio.

Silvers, James L., Trent Tile Co., Inc., Trenton, N. J.

Taylor, R. F., Pilkington Bros., Ltd., Niagara Falls, N. Y.

Corporation.

Beaver Falls Art Tile Co., Beaver Falls, Pa.

Laclede-Christy Clay Products Co., St. Louis, Mo.

Midland Terra Cotta Co., 115 Lumber Exchange Bldg, Chicago, Ill.

NECROLOGY.

Owen Carter, of Poole, England, was educated in private schools and on leaving entered the encaustic tile business of Carter and Company, which had been established by his father about five years before. The first few years of his business career were spent at the London office of the firm, but he subsequently went to Poole and undertook the works management which he retained to the time of his death, in February, 1919.

Mr. Carter was deeply interested in scientific subjects, particularly those bearing upon the practical side of the business, and his keen appreciation of art did much to influence the artistic production of the firm. The business, which was established in quite a small way in 1873, grew to be one of the most important in England, comprising three large factories doing a considerable home and export trade in floor and wall tile, constructional faience and terra cotta, mosaic, fire-places, fancy pottery, etc. The development of these various branches were largely due to Mr. Carter's initiative and practical skill. Of a very retiring disposition, Mr. Carter never took active part in public life but devoted his energies to business and to "doing his bit" in a quiet, unostentatious way.

Mr. Carter was a member of the Society of Friends and was faithful to the traditions of that body in the simplicity and honesty of his life. He was elected to Active Membership in the American Ceramic Society at the last annual meeting, but he did not live to learn of this. He died in his fifty-seventh year, after an illness extending over four months.

Hugo J. Lundgren was born in Sweden, in 1881, and was educated partly in his native country and partly in Germany. He entered the employ of the Canadian General Electric Company, at Petersborough, Ontario, in 1913 and was continuously engaged with this concern until his death.

He specialized in electrical porcelain and succeeded in bringing his department to a high degree of excellence. He was attacked

by the influenza epidemic during a professional visit to various porcelain factories and reached his home in an advanced stage of sickness. He died February 24, 1919, at the age of thirty-eight.

Mr. Lundgren was elected to Active Membership in the American Ceramic Society at the Pittsburgh meeting but did not live to receive the notice of this. He was a member of the American Institute of Electrical Engineers, before the Toronto branch of which he read last winter a valuable paper on porcelains for electrical purposes.

William A. Kemp, of Toronto, Canada, died suddenly on March 18, 1919, at Pine Forrest Inn, Summerville, South Carolina. His health had never been robust since an attack of typhoid fever, contracted when he was traveling in Italy some years ago, and he had gone south to try to regain it.

Mr. Kemp was born in Clarenceville, Quebec, fifty-six years ago and went to Toronto in 1886 to engage in business with his brother under the name of the Kemp Manufacturing Company, which afterwards developed into the Sheet Metal Products Company of Canada, Limited. Mr. Kemp was the vice-president of that Company and was also president of the MacDonald Manufacturing Co. of Toronto, Limited. Through his experiments and interest the enamel ware industry was founded in Canada, replacing the German and Austrian products which then supplied the market.

Mr. Kemp was a man of sagacity, sympathy, and friendliness and was generous in the use of his means. He was particularly interested in all sorts of work for young men and boys, and in the missionary enterprises of the Methodist denomination of which he was a member. His family consists of his widow, three daughters and one son, Captain Clarence Kemp, who is still in France with the Canadian Expeditionary Forces.

A. Lawrence Jensen, of Brooklyn, New York, died at the Hotel Hollywood, Southern Pines, N. C., on February 14, 1919. Mr. Jensen was one of the directors of the Empire China Works, Brooklyn, and had been connected with the porcelain industry since 1890.

Mr. Jensen is survived by his wife, two daughters, a sister, and a brother, Chas. H. Jensen, an Active Member of the Society.

ACTIVITIES OF THE SOCIETY.

The following motions have recently been passed by the Board of Trustees:

April 8th. It was voted that all discussions and votes by members of the Board of Trustees be directed to the President, as chairman of the Board, and that copies be sent to all other members of the Board when such are mailed to the Secretary by each member. It was voted that in case the President does not wish to second a motion he immediately notify the maker who shall secure a seconder.

The appointment of Professor A. V. Bleininger as representative of the American Ceramic Society on the Division of Chemistry and Chemical Technology of the National Research Council was confirmed by the Board.

April 10th. It was voted to hold the Summer meeting, 1919, in Buffalo, Niagara Falls, and Cleveland, the meeting to convene at the Hotel Statler, Buffalo, on Monday evening, August 4th.

April 14th. It was voted to give a complimentary subscription to the JOURNAL of the Society to the Library of the Chemists' Club, New York City.

April 18th. It was voted to hold the Twenty-second Annual Meeting of the Society at Philadelphia, February 23–26, 1920, with headquarters at the Hotel Walton.

April 24th. On the recommendation of the Committee on Papers and Programs, it was voted that the papers that may have been published in the JOURNAL previously to the Annual Meeting shall appear on the printed program, and their authors shall be given the privilege of presenting them on the floor at the meeting; that the program shall announce the names of those who have signified a desire to discuss a paper; and that the Committee shall demand that copies, or at least abstracts, of all papers be in the hands of the Committee by February 1st, unless previously printed.

April 24th. It was voted to nominate Dr. Robert B. Sosman and Mr. H. F. Staley as representatives of the Society upon the Committee on Ceramic Research, National Research Council (see editorial in March number).

Action of the Society.

April 18th. It was voted, by letter ballot, to adopt the Amendments to the Rules, proposed by the Committee on Rules at the Annual Meeting, Pittsburgh, 1919.

ACTIONS TAKEN BY THE BOARD OF TRUSTEES DURING THE YEAR 1918-1919.

At a meeting of the Board of Trustees at Pittsburgh, it was voted to publish monthly the resolutions and motions passed by the Board, so that the members of the Society may be apprised of the actions taken from time to time.

The following is a summary of the motions passed during the year 1918–1919:

Professor G. H. Brown was elected Editor and Miss M. M. Gill as his assistant. Miss Norah W. Binns was appointed assistant to Professor Chas. F. Binns, the Secretary. Early in the year the Editor was empowered to submit a monthly bill for office expense, and later the President was empowered to submit a bill for stenographic assistance. The sum of $100 was appropriated for the expenses of the booth of the Society at the Chemical Exposition. Twice during the year it was found possible to invest some of the Society's funds. It was voted to invest $3,000 in the Fourth Liberty Loan and $2,000 in U. S. Treasury Certificates.

The Committees on Coöperation and Local and Student Sections were appointed by the President and approved by the Board. Mr. A. F. Greaves-Walker was appointed chairman of the War Service Committee and Mr. R. C. Purdy, chairman of the Membership Committee.

The Secretary was empowered to accept applications for Associate and Contributing Membership for one year. The seventeen Associates nominated to Active Membership at the Annual Meeting (1918) were voted upon and elevated.

When the JOURNAL was launched it was found that the postal regulations necessitated a change in the wording of the Rules, with regard to the subscription price to members. This change was approved by the Board of Trustees and a resolution was passed by the Society at the Summer Meeting.

The Board voted to place fifty copies of each issue of the JOURNAL at the disposal of the Membership Committee for the purpose of soliciting new members.

A discount of 20 per cent to dealers upon the subscription price of the JOURNAL was decided upon.

At the Summer Meeting the Society voted to support Mr. A. F. Greaves-Walker, chairman of the Industrial Furnace Section of the U. S. Fuel Administration, by assisting in the securing of volunteer inspectors. The Board voted to give space in the JOURNAL to advertise for these.

The methods of formation of Professional Divisions was decided upon as follows:

I. "A petition may be presented by not less than ten members in good standing, of whom three or more shall be Active, who are interested especially in some phase of ceramic work sufficiently broad to warrant the formation of a special Division. This petition shall go to the President, who shall then appoint a representative committee to consider the advisability of forming such a Division, and to proceed with the organization if the decision is favorable."

II. "When the initiative is taken by the Board of Trustees in order to stimulate the growth of the Society, the President shall appoint a representative man to furnish the initiative. He shall select his own committee." It was later voted that Divisions on Glass, Enamel, Pottery and Porcelain, Refractories, Brick and Tile, Terra Cotta and Faience, and Abrasives, should be established.

Pittsburgh, Pa., was chosen as the place of the Twenty-first Annual Meeting.

The Board voted to advertise the "Directory of Dealers" in two successive issues of the JOURNAL.

The stock of certain volumes of the Transactions having become low, it was voted to purchase a sufficient number of these to complete fifty sets, which should then be held for sale in sets only.

It was voted that 200 copies of each number of the JOURNAL be reserved at the time of issue for sale in complete sets only, and that an additional 200 be likewise reserved for sale in complete volumes only. It was the sense of this resolution that a "complete set" should consist of the first five volumes of the JOURNAL and such additional volumes as may have been issued up to the date of the sale, and that a "sale" should be the actual selling of volumes and not the furnishing of them in consideration of payment of membership dues. It was voted at the same time that copies of the volumes of Transactions and of the JOURNAL be furnished to members who have allowed their dues to lapse, only after the number reserved for sale has been provided. Furthermore, that in cases in which the volume for a certain year's dues is not available, the member shall have the option of selecting any available volume or having the dues apply to the succeeding year.

The board voted that, beginning with Volume 2, Number 1, authors of papers published in the JOURNAL shall be furnished with reprints, free of charge, in any number, not to exceed 100, which shall be requested.

CHAS. F. BINNS, Secretary.

LOCAL SECTIONS.

Chicago Section.

The Sixth Meeting of the Chicago Section of the American Ceramic Society was held in connection with the Forty-first Annual Convention of the Illinois Clay Manufacturer's Association, at the La Salle Hotel, Chicago, April 9, 1919. After an appropriate talk by Mr. W. D. Gates, acting as Chairman *pro tem*, the following papers were presented:

"Discussion of the Humidity System of Drying Clay Wares," by Mr. B. S. Radcliffe, Midland Terra Cotta Co., Chicago.

"Heat Insulation for Industrial Furnaces," by Mr. H. N. Haberstroh, Celite Products Co., Chicago.

"Notes on Zirconium," by Mr. E. E. Libman, University of Illinois, Urbana, Illinois.

Mr. F. B. Ortman joined in the discussion of humidity drying and contributed ably to this interesting subject. All papers were enthusiastically received and discussed.

Professor C. W. Parmelee enlightened the Society on the present status of the Ceramic Department at the University of Illinois, and informed the members that the Board of Trustees had authorized the Chicago Section to nominate four members as its representatives on the Advisory Committee of the Ceramic Department. The following men were unanimously appointed by the Section:

Mr. E. F. Achardt, General Manager, Federal Plate Glass Co., Ottawa, Ill.

Mr. Chas. S. Reed, President, Chicago Retort and Fire Brick Co., Ottawa, Ill.

Mr. Theo. G. Dickinson, President, Marquette Cement Co., Chicago, Ill.

Mr. F. H. Fall, Jr., Benjamin Electric Co., Chicago, Ill.

It was voted that the Chair appoint one member to coöperate with representatives of the Societies listed below, in fostering the close and lasting affiliations of these Societies.

Illinois Clay Manufacturer's Association.
Illinois Paving Brick Manufacturer's Association.
Illinois, Indiana Division, American Face Brick Association
Indiana Brick Manufacturer's Association.
Illinois Drain Tile Manufacturer's Association.
American Ceramic Society.

Adjourned.

H. T. BELLAMY,
Secretary-Treasurer.

New York State Section.

The New York State Section of the American Ceramic Society held a meeting in Syracuse, N. Y., Friday and Saturday, May 9th and 10th, at which the following program was presented:

Friday, 10 A.M., Business meeting of the Section.

At the Friday afternoon session the following interesting papers were presented:

"Address of Welcome," by B. E. Salisbury, Onondaga Pottery Co.

"Mechanical Phases of Ceramic Engineering: Drying and Firing," by L. E. Barringer, General Electric Co.

"The Manufacture of Abrasive Wheels," by S. C. Linbarger, Carborundum Company.

"The Porcelain Situation in the United States," by Prof. C. F. Binns, Alfred University.

"Fuel Regulation in Ceramic Plants," by Prof. G. A. Bole, Alfred University.

"Some Observations on the Shrinkage of China Bodies," by Mark A. Haley, Onondaga Pottery Co.

"How to Make Enamels Durable," by Prof. J. B. Shaw, Alfred University.

On Saturday parties visited the Onondaga Pottery and the Paragon Plaster Company.

The meeting was very well attended, there being members present from every section of the State. In addition to the members of the New York State Section the session was attended by brick manufacturers of western New York.

At the business meeting much business was transacted, and among other things it was decided to call a meeting of the New York State Section at Buffalo, at the time of the August meeting of the Society. The officers elected for the current year were as follows:

Chairman—S. C. Linabarger.
Secretary-Treasurer—J. B. Shaw.
Councilor—L. E. Barringer.

Adjourned.

J. B. Shaw.
Secretary-Treasurer.

Northern Ohio Section.

The fifth meeting of the Northern Ohio Section of the American Ceramic Society was held at the Portage Hotel, Akron, Ohio, on April 28th.

In the morning a visit was made to the plant of the Goodyear Tire and Rubber Co. After lunch at the hotel a trip was made through Plant No. 1 of the Robinson Clay Products Co. This plant is devoted to the manufacture of stoneware, including large and intricate chemical shapes.

A short business meeting preceded the program. The first paper of the afternoon was given by Prof. Hewitt Wilson, Ohio State University, on "Glaze Studies." This was followed by a talk on "The Present Trend in Porcelain Manufacture," by Mr. E. T. Montgomery of the Jeffery-Dewitt Co., Detroit, Mich.

Mr. William P. Blair, Secretary of the National Paving Brick Manufacturers' Association, gave a very interesting talk dealing with present day requirements for both art and utility in all kinds of brick construction.

The program closed with an illustrated talk by Mr. E. P. Poste, Elyria Enameled Products Co., Elyria, Ohio, on "The Manufacture of Enameled Ware."

The attendance was good in spite of the bad weather.

Adjourned.

B. A. Rice,
Secretary-Treasurer.

Pittsburgh District Section.

A meeting of the Pittsburgh District Section of the American Ceramic Society was held at the Potter's Club, East Liverpool, Ohio, on Wednesday evening, April 23, 1919.

Mr. W. E. Wells, of the Homer Laughlin China Co., presented an interesting discussion of "Cost Accounting in Pottery Manufacture."

Mr. F. W. Walker, Sr., of the Beaver Falls Art Tile Co., Beaver Falls, explained some of the work which has been done recently in Washington in establishing prices for various clay products.

The meeting was very well attended and thoroughly enjoyed by all.

F. H. RIDDLE,
Secretary-Treasurer.

Adjourned.

Eastern Section.

The New Jersey Clay Workers Association and Eastern Section of the American Ceramic Society will hold a meeting at the Trenton Country Club, Trenton, N. J., on Tuesday, June 24th. The usual excellent program is being arranged for the morning and afternoon sessions of this meeting. Members of the Society are cordially invited to attend, particularly those residing in New Jersey, New York City, Eastern Pennsylvania, Eastern Maryland and Delaware.

Applications for membership in the Eastern Section should be forwarded to G. H. Brown, Box 444, New Brunswick, N. J

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EDITORIALS.

THE SUMMER MEETING.

As previously announced in these columns, the 1919 Summer Meeting of the American Ceramic Society will be held at Buffalo, Niagara Falls and Cleveland, August 4th to 8th, inclusive, the meeting to convene at the Hotel Statler, Buffalo, on Monday evening, August 4th. As usual the daytime will be spent in inspection trips to plants of ceramic interest in the vicinity of the above cities—the evenings being devoted to such business meetings and entertainments as are being arranged by the Committee on Summer Meeting of the Society and the local entertainment committees.

The Refractories Division announces a meeting to be held at Niagara Falls on August 4th, at which several important matters dealing with the future work of the Division will be discussed. Meetings of other Divisions will be held during the week. Cooperating with the Summer Meeting Committee, the Northern Ohio Section is arranging an itinerary and entertainment for the visiting members of the Society while in Cleveland and vicinity.

The central location, excellent rail and boat facilities, and the many ceramic plants of interest in the Buffalo and Cleveland districts should assure an excellent attendance at this meeting. Aside from the opportunity to visit the modern pottery, glass and enameling plants at Buffalo and Cleveland, the electrochemical plants at Niagara Falls should prove of unusual interest to our members.

Full details of the arrangements for the meeting will be announced in the June number of the Journal.

PROFESSIONAL DIVISIONS.

It is gratifying to note that the Terra Cotta Division of the American Ceramic Society is now fully organized and that considerable progress has been made in outlining and getting under way the work for this year. Enrolled in this Division are forty-nine members representing twenty-four terra cotta companies, the Bureau of Standards, and the Ohio State University.

Both the Ohio State University and the Bureau of Standards are now doing research work on terra cotta bodies and are willing at all times to coöperate with members of the Division in solving any of their problems and in conducting the investigations which have been undertaken by the members of the Division in their own laboratories and plants. These investigations cover a wide field including: the improvement and development of glazes; the effect of electrolytes on terra cotta bodies; the effect of the size of grog in terra cotta bodies; spalling of terra cotta bodies; spraying machines; humidity drying; firing terra cotta kilns; standardization of terra cotta colors; etc.

It is the intention of the officers of the Division to have the results of as many of the above investigations as possible prepared for submission to the members of the Division some time before the next annual meeting of the Society, in order to allow a free discussion before their formal presentation before the Society.

The terra cotta industry should be well pleased with the progress made in the last few months in technical coöperation between the companies and the American Ceramic Society and with the interest shown in getting some valuable research work started which is bound to result in improved methods of manufacture, better terra cotta and fewer failures. The formation of the Terra Cotta Division at the last annual meeting has already been more than justified.

When the rapidly maturing plans and investigations of the other Divisions of the Society are well under way, there will be no question as to the value of group organizations of this kind in the technical advancement of the different branches of the ceramic industry and in adding to the technical literature and membership of the Society.

AMERICAN ENGINEERING STANDARDS ASSOCIATION.

The American Engineering Standards Committee has just completed a revision of its Constitution which has been sent to the Governing Boards of all the Departments and Societies represented on the Committee with a request for its ratification. The new Constitution changes the name to American Engineering Standards Association, the change from "Committee" to "Association" more fittingly indicating the wide scope of the interests involved.

Included in the Founder Societies of the Committee were the American Society of Civil Engineers, American Institute of Mining Engineers, American Society of Mechanical Engineers, American Institute of Electrical Engineers and the American Society for Testing Materials. The objects of the Association are stated as follows:

1. To unify and simplify the methods of arriving at engineering standards, to secure coöperation between various organizations and to prevent duplication of standardization work.

2. To promulgate rules for the development and adoption of standards.

3. To revise and pass upon recommendations for standards submitted as provided in the Rules of Procedure, but not to initiate, define or develop the details of any particular standard.

4. To act as a means of intercommunication between organizations and individuals interested in the problems of standardization.

5. To give an international status to approved American engineering standards.

6. To coöperate with similar organizations in other countries and to promote international standardization.

In view of the importance of standardization in the testing and classifying of ceramic materials and products and of the extensive work now being carried on by the Committee on Standards of the American Ceramic Society, it would appear that the Society should be vitally interested in furthering the purposes of the Standards Association. In the past there have been duplications and wasted efforts in attempts to standardize tests for ceramic materials. There should be technical coöperation without as well as within the Society in arriving at suitable standards.

ORIGINAL PAPERS AND DISCUSSIONS

A STUDY OF SOME LIGHT-WEIGHT CLAY REFRACTORIES.

By M. F. Beecher.

The work described in this paper was suggested by the investigation on light-weight refractories promoted by the Pittsburgh Laboratory of the Bureau of Standards and conducted by them in coöperation with the Navy Department. Their development of a brick of attractive quality has been the incentive inspiring others to a similar line of work, and enough interest is being taken in this new product to indicate that it may have a considerable field of usefulness.

It is not the purpose here to bring out the advantages or properties of specific composition, so much as to show by means of the data presented some of the factors involved and some of the limits met in the making of light-weight clay refractories. The data are not sufficient, either in volume or detail, to cover the subject thoroughly, but the comparative nature of most of the results will indicate the general quality of brick which may be produced.

The Bricks Tested.—In this study, bricks of 12 different compositions were subjected to test. The first seven of these—those upon which our attention will be particularly centered—were experimental light-weight bricks. The other five were full-weight commercial bricks tested to supply a basis for comparison. The compositions appear in Table 1, the parts being by volume.

TABLE 1.—COMPOSITIONS OF THE BRICKS TESTED.

No.	Lab. No.	No. 1 refractory clay. Per cent.	Kaolin. Per cent.	Regular grog. Per cent.	Kaolin grog. Per cent.	Sawdust. Per cent.
1....	M84	19	38	24	..	19
2....	M86	10	30	13 1/3	..	46 2/3
3....	M87	50	10	....	10	30
4....	M88	50	10	10	..	30
5....	M89	50	10	20	..	20
6....	M91	50	20	....	..	30
7....	M94	25 1/3	24 2/3	....	..	50
8....	M77	Norton special clay brick.			..	..
9....	M22	A good quality silicious clay brick made in New England of New Jersey clays.				
10....	M85	A Pennsylvania flint-clay brick.				
11....	M90	A Missouri flint-clay brick.				
12....	M95	A Kentucky flint-clay brick.				

Each of the last four commercial fire bricks described above represents approximately the best quality produced in each district. The grog referred to as regular grog is that which is produced in reclaiming broken slabs and saggers. It contains, of course, no slag or fusible material of any kind. The kaolin grog was prepared by calcining washed Florida kaolin at cone 13. In mixtures of this kind the greatest variation is likely to be found in the kind or character of sawdust used. The sawdust in these bricks contained no hard woods. It had a weight of 14 pounds per cubic foot when shaken down slightly and was sized to pass an 8-mesh screen.

The bricks were made by hand in single wooden molds from a mixture thoroughly pugged in a small auger machine. The manufacturing data appear in Table 2 following:

TABLE 2.—MANUFACTURING DATA.

No.	Lab. No.	Workability.	Drying behavior.	Drying shrinkage.	Burning shrinkage.	Total shrinkage.
1....	M84	Sl. short	Good	4.7	4.6	9.1
2....	M86	Short	Good	4.4	7.8	11.4
3....	M87	Good	Good	4.0	3.8	7.6
4....	M88	Good	Good	4.8	3.2	7.8
5....	M89	Good	Good	4.0	3.2	7.1
6....	M91	Sl. short	Good	5.1	3.7	8.6
7....	M94	Short	Good	5.1	7.2	11.9

Cold Physical Properties.—A more complete description of the bricks is provided by the data of Tables 3 and 4 which follow. The cold physical properties given in Table 3 were obtained by the usual methods. They consisted of the determination of the modulus of rupture, porosity, apparent specific gravity, bulk specific gravity, and the weight reduced to the standard $2^1/_2'' \times 4^1/_2'' \times 9''$ size.

TABLE 3.—COLD PHYSICAL PROPERTIES OF THE BRICKS TESTED.

No.	Lab. No.	Modulus of rupture, lbs. per sq. in.	Per cent porosity.	Bulk sp. gr.	Apparent sp. gr.	Wt. per std. $2\frac{1}{2}'' \times 4\frac{1}{2}'' \times 9''$, pounds.
1.....	M84	716.0	39.5	1.54	2.57	5.51
2.....	M86	270.0	56.5	1.11	2.59	4.01
3.....	M87	547.0	47.7	1.46	2.80	5.43
4.....	M88	474.0	46.1	1.50	2.80	5.50
5.....	M89	436.0	43.0	1.60	2.81	5.84
6.....	M91	533.0	51.8	1.48	3.06	5.35
7.....	M94	283.0	60.6	1.10	2.81	3.93
8.....	M77	558.0	31.3	1.87	2.72	6.76
9.....	M22	304.0	32.0	1.82	2.65	6.58
10.....	M85	436.0	21.0	1.94	2.46	6.87
11.....	M90	...	17.4	2.17	2.63	7.92
12.....	M95	457.0	22.1	2.09	2.68	7.43

The modulus of rupture was calculated by the usual formula $R = \frac{3\ Pl}{2\ bd^2}$ from the transverse breaking load on five specimens. The breaking machine was of simple construction, having a lever ratio of 1 to 20. The knife edges were of steel and rounded to a radius of $^1/_4''$. One was fixed and the other two free to move, thus allowing for any unparallel condition of the specimens. The load was applied by means of falling shot and the machine load measured was $^1/_2$ P.

The porosities were determined upon half-bricks resulting from the transverse breaking test. The dry weights, saturated weights and suspended weights were obtained using distilled water as the saturating and suspending medium. Saturation was accomplished by boiling submerged for one hour and the calculation was by Purdy's formula $\frac{W - D}{W - S} \times 100 =$ per cent porosity.

The specific gravities were obtained from the same values by the relations $\frac{D}{D - S}$ = apparent specific gravity, and $\frac{D}{W - S}$ = bulk specific gravity.

The weights reduced to standard dimensions were obtained by calculations from actual measurements and weights.

TABLE 4.—AVERAGE WEIGHTS OF COMMERCIAL FIRE BRICKS.

Source.	Number of brands.	Maximum weight, pounds.	Minimum weight, pounds.	Average weight, pounds.
Pennsylvania.....	11	8.22	6.97	7.46
W. Virginia.....	1	..	..	7.58
Illinois.....	2	7.42	6.89	7.15
Maryland.....	6	8.11	7.05	7.34
Missouri.....	7	7.81	7.22	7.56
Kentucky.....	2	8.55	8.14	8.34
Colorado.....	1	..	..	7.44
Ohio.....	7	7.68	6.56	7.14

Table 4 above presents some data on the weights of commercial fire brick which will be of interest in connection with the data of Table 3. Of the 37 different brands of commercial fire brick represented here the average weight was found to be 7.46 pounds. In general, there appears to be a very wide variation in the weights of fire bricks from the same locality; too much importance therefore should not be placed upon these averages resulting from a very small number of bricks—for instance, the average of 8.34 pounds for Kentucky brick, where only two different brands were available, is hardly representative.

Chemical Analyses.—In Table 5 appear the chemical analyses of these bricks. In several cases the mixtures differed only in the quantity of sawdust present and only one analysis, therefore, was made to cover such cases.

It is not expected that any deduction as to the quality of these bricks can be made from these analyses, but they will show what wide variations in chemical composition are possible between bricks of equal or similar ultimate quality.

TABLE 5.—CHEMICAL ANALYSES OF THE BRICKS TESTED.

No.	Lab. No.	SiO_2.	Al_2O_3.	Fe_2O_3.	TiO_2.	CaO.	MgO.	K_2O and Na_2O.	Total fluxes.
1	M84	...	...	..	..	..	..	..	..
2	M86	62.66	33.96	0.46	1.70	0.54	0.62	0.28	1.90
3	M87	...	...	..	..	..	..	..	..
4	M88	41.84	52.77	1.11	3.10	0.32	0 70	0.40	2.53
5	M89	44.52	50.31	0.51	3.50	none	0.73	0.30	1.54
6	M91	28.08	64.21	2.91	4.10	none	0.67	0.28	3.86
7	M94	...	...	..	..	..	..	..	..
8	M77	45.64	48.09	2.07	2.20	0.56	0.55	0.56	3.74
9	M22	80.02	13.86	2.72	1.70	0.36	0.63	0.62	4.33
10	M85	50.24	43.18	1.94	3.00	0.44	0.81	0.32	3.51
11	M90	57.14	37.55	1.83	1.60	0.56	0.77	0.54	3.70
12	M95	58.64	35.22	2.64	2.50	0.08	0.77	0.22	3.71

Load Carrying Capacity at Elevated Temperatures.—The load test method conformed to the A. S. T. M. tentative standard.[1] A load of 50 pounds per square inch was used because a considerable amount of data obtained from tests under those conditions was already available for comparison, and also because the greater load would allow of greater deformation thus serving to differentiate more closely between the bricks of highest quality. In Table 6 following are presented the data resulting from this test:

A discussion of these results[2] has brought out that in comparison with tests on the same brands of commercial brick in another laboratory, the high deformation here noted might indicate that the test conditions applied were somewhat severe. However, very close attention was given to the set-up and manipulation. The thermocouple cold-junction was maintained between 20° and 25° C throughout the test. It was learned later[3] that the commercial brick represented by No. 12 did not appear to be as dense or as well burned as those ordinarily produced by this manufacturer. We, therefore, can account for this apparent variance of results in the possible difference in quality of bricks of the same brand[4] and the possible use of different types of testing

[1] *Proc. A. S. T. M.*, **17**, Part 1, p. 665 (1917).

[2] Private correspondence.

[3] Information from an extensive user of that brand.

[4] *J. Am. Ceram. Soc.*, Vol. 1, p. 403. Loomis mentions this possibility.

TABLE 6.—RESULTS OF LOAD TESTS.

50 pounds per sq. in. at 1350° C.

No.	Lab. No.	Initial length, inches.	Final length, inches.	Deformation.		Remarks.
				Inches.	Per cent.	
1...	M 84	9.03	8.79	0.24	2.66	.....
2...	M 86	8.80	7.87	0.93	10.57	.....
3...	M 87	9.25	Shear	..	...	45 minutes at 1350° C.
4...	M 88	9.19	8.68	0.51	5.54	.....
5...	M 89	9.28	9.06	0.22	2.37	.....
6...	M 91	9.21	8.79	0.42	4.56	.....
7...	M 94	8.85	6.74	2.11	23.80	30 minutes at 1350° C.
8...	M 77	9.07	8.84	0.23	2.54	.....
8...	M 77	9.08	8.91	0.17	1.87	.....
9...	M 22	9.01	8.88	0.13	1.33	.....
10...	M 85	9.22	8.57	0.65	7.06	.....
11...	M 90	8.78	8.04	0.74	8.43	.....
12...	M 95	8.86	7.23	1.63	18.40	.....

furnaces in the two laboratories. In any event it is evident that the results presented in Table 6 are comparable within this study.

Fig. 1 shows the manner off ailure of the bricks tested. The photographs exhibit an interesting feature, namely, the absence of serious buckling of the specimens. Even where the deformation is very great, as in Nos. 7 and 12, the buckling was not sufficient to have been a serious factor in the deformation.

Spalling Behavior.—The spalling test followed somewhat the method of Nesbitt and Bell,[1] except that the cooling was accomplished in air instead of in water. The heating was done in the standard load-test furnace. The heating chamber was filled with a checker-work of fire brick, set on edge, built up two high. The thermocouple hot-junction was within 1″ of the lower end of the test bricks and approximately in the center of the furnace. Since the capacity of the furnace was eight bricks, two runs were necessary, but care was taken to see that the same rate of heating and the same cooling intervals were followed in both runs.

The furnace as described, with the test bricks in place, was heated up to approximately 1300° C and the bricks then with-

[1] *Proc. A. S. T. M.*, 16, part 2, p. 349.

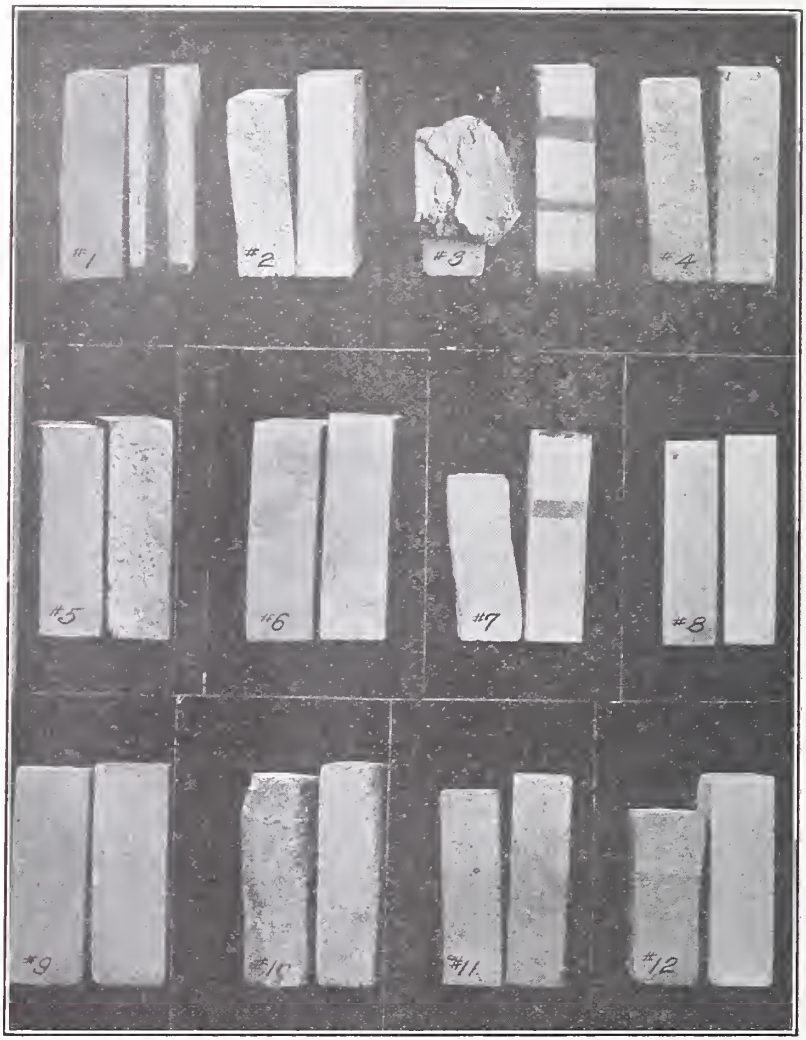


FIG. 1—Behavior of bricks in load test.

drawn one at a time and cooled in a blast of air for exactly one minute. The air issued from a nozzle, $^{5}/_{8}''$ in diameter and $4''$ from the bricks, under a pressure of five pounds. The blast was directed at right angles against the hot end. This was repeated

every 20 minutes—since that was the time necessary for the furnace temperature to return to approximately 1300° C. The test was continued to 30 heatings and coolings.

The operation of removing from the furnace and returning the cooled brick to the furnace required on the average of 15 seconds. During the first ten minutes, therefore, of the 20 minute cycle there were only seven bricks continuously in the furnace and the temperature would drop from 1300° to 1250° C. During the last ten minutes of the cycle the eight bricks would be in place and the temperature would return to 1300° C again by the next cooling period. Notes on the development of cracks and spalls were taken during the test—for many cracks that were very apparent while the brick was under the air blast could not be distinguished when the brick had finally cooled.

In Fig. 2 are shown, graphically, the results obtained. While Nesbitt and Bell[1] have measured the spalling by the loss in weight of the bricks at the conclusion of thirty coolings, this did not appear to afford a good means of judging the results in the present instance. Many of the bricks showed no loss in weight whatever but these could be quite readily classified on the basis of the extent of cracking. Thus we have indicated as accurately as possible the extent and position of the cracks in one face and in the heated end of each brick, after the thirtieth cooling, or the discontinuance of the test. The double line indicates a complete fracture or spall while the single line represents only a crack. The small number in the upper right hand corner of each brick indicates its rank as determined by this test, brick marked "1" showing the greatest resistance to spalling.

Nor is this method of classification found entirely satisfactory. No. 2 and No. 7 have been ranked 12 and 11, respectively, while it is entirely probable that the fractures produced during the test were not due to the repeated heating and cooling as much as to the rather rough handling which they of necessity received. These two bricks, it will be observed upon reference to Table 3, have very low mechanical strength and the effect of the handling would naturally be exaggerated in proportion. Brick No. 3

[1] *Loc. cit.*

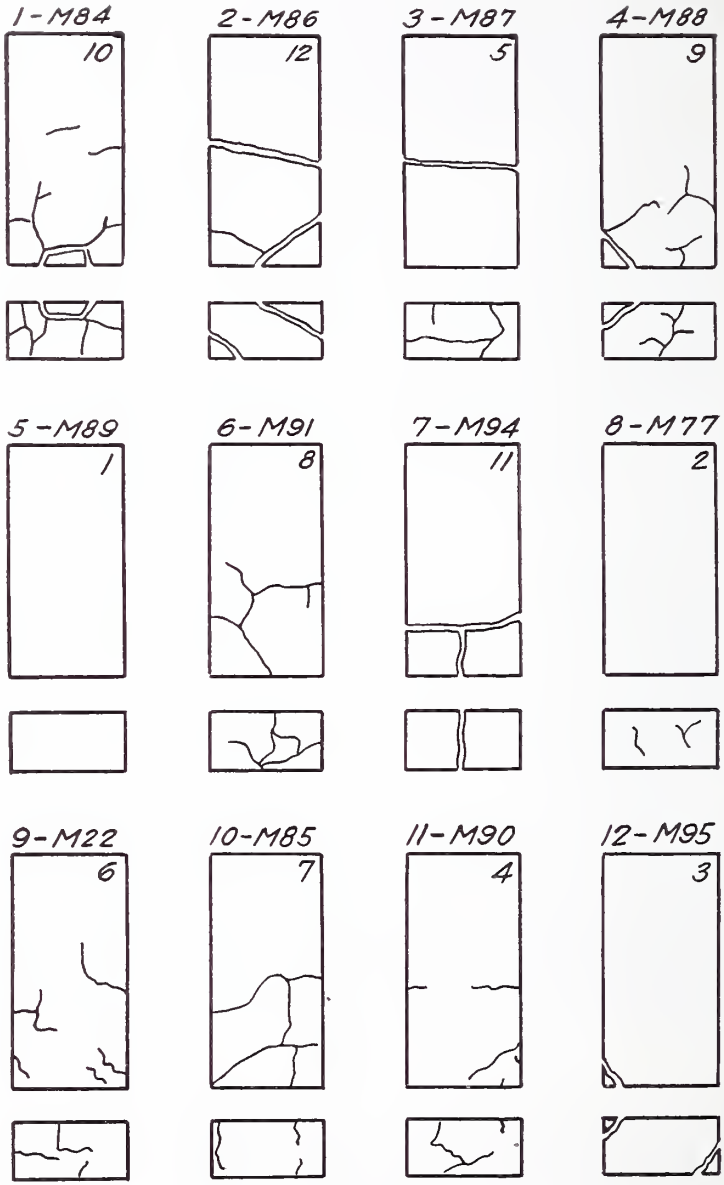


FIG. 2.—Results of spalling tests.

showed very few cracks after the test, but on removing from the furnace after the thirtieth heating, it was found to be in two pieces. This case also, we are satisfied, can be attributed to the low mechanical strength of this mixture.

Fusion Point.—The fusion point determinations were made through the courtesy of Mr. R. H. White.[1] The test cones were made by crushing portions of each of two bricks, amounting to approximately one-quarter of a brick, between steel surfaces to pass a 60-mesh screen. An addition of gum was then made and cones of the standard dimensions molded in a brass mold. These were set up with standard cones on refractory pats made of a mixture consisting of 85 per cent of fused alumina and 15 per cent of refractory bond clay. The cone pats were $2\frac{1}{4}''$ wide and $5''$ long, and each contained two rows of cones of seven cones each. In each row the four standard cones were alternated with three test cones. The cones in the two rows were placed in reverse order, thus preventing any unequal heating of the furnace from affecting the results.

In Table 7 are given the results obtained in the fusion tests:

TABLE 7.—RESULTS OF FUSION TESTS.

Number.	Laboratory number.	Cone of fusion.
1.....	M84	31–32
2.....	M86	31–32
3.....	M87	31–32
4.....	M88	31–32
5.....	M89	31–32
6.....	M91	32
7.....	M94	32
8.....	M77	32
9.....	M22	29–30
10.....	M85	32
11.....	M90	31–32
12.....	M95	Not made

Thermal Insulation.—Only a simple and approximate test was made to determine the relative behavior of the bricks as regards

[1] Tech. Director, Abrasive Plants, Norton Company, Niagara Falls, N. Y.

thermal insulation. It is not intended that the results presented should be considered as accurate or even very exact measures of the insulation properties. The factors involved in an exact thermal study are so many and varied and the importance of very careful manipulation so great that there was neither the time, nor in fact, the necessity for it in the present instance. However, the information conveyed in the curves of Fig. 3 shows roughly the relative behavior of the bricks as insulators.

These data were obtained by subjecting one face of the test specimen to the direct flame of a gas burner and noting the temperature rise per time of the opposite face. The temperature of the flame was constant at 1100° C ± 15°. The set-up was exactly the same for every test and was protected against variable air currents. Readings were taken at five-minute intervals for one hour or until the cooler face reached a temperature of 350° C.

For purposes of comparison, a Silocel insulating brick and a Crystolon brick were subjected to the same test and the curves plotted as for the others.

While the differences between the bricks are not especially marked, they are sufficient to differentiate roughly between the good and the poor The experimental clay bricks average up equal or superior to all the commercial brands except No. 12.

Summary.

For convenience the major results from each of the preceding tests are assembled in Table 8. These show that, except for Nos. 2 and 7, the load-carrying capacities compare very favorably with the standard full-weight bricks. In resistance to spalling the light-weight bricks again show up very well. In thermal insulation they are practically equal to or superior to all the commercial brands except No. 12.

The weights of the experimental bricks are somewhat parallel with the porosities, though the character of the clays used may have a marked influence on weight.

The porosity is governed by the character of the clays, the amount and size of the grog, and the amount of sawdust added. The more open-burning clays and the more uniformly sized grog

TABLE 8.—SUMMARY OF DATA FROM COMPARATIVE TESTS.

No.	Weight		Load Test.		Resistance to spalling.		Cone of fusion.	Thermal insulation. Rank.
	Rank.	Standard $2\frac{1}{2}'' \times 4\frac{1}{2}'' \times 9''$.	Rank.	Per cent deformation.	Rank.	Behavior.		
1..	6	5.51	4	2.66	10	fair	31–32	10
2..	2	4.01	9	10.57	12	poor	31–32	3
3..	4	5.43	12	Shear	5	good	31–32	5
4..	5	5.50	6	5.54	9	fair	31–32	6
5..	7	5.84	3	2.37	1	good	31–32	2
6..	3	5.35	5	4.56	8	fair	32	4
7..	1	3.93	11	23.80	11	poor	32	8
8..	9	6.76	2	2.19	2	good	32	11
9..	8	6.58	1	1.33	6	fair	29–30	9
10..	10	6.87	7	7.06	7	fair	32	7
11..	12	7.92	8	8.43	4	good	31–32	12
12..	11	7.43	10	18.40	3	good	...	1

will naturally make the more porous brick. Sawdust additions (to relatively open-burning clay mixtures) appear to increase the porosity by an amount equal to the actual volume of the addition. Thus, comparing M77 and M88, which are very similar in clay content, we find the porosities to be 31.3 per cent and 46.1 per cent with sawdust contents of 0 and 30 per cent, respectively. The actual volume of this sawdust is about one-half its bulk volume, or, the wood particles added actually constitute but 15 per cent. The difference in the porosities is 14.8 per cent. M91 and M94 have porosities of 51.8 per cent and 60.6 per cent with a difference in sawdust content of 20 per cent. The difference in porosity is 8.8 per cent.

The *load carrying capacity* of fire bricks at elevated temperatures, it has been observed, seems to have no relation to the strength in the cold condition.[1] When the compositions are similar, however, and the porosity is varied by induced methods, the deformation varies with the porosity or openness. Examples are those of M87, M88 and M89.

The results obtained on the light-weight bricks show that all

[1] A. V. Bleininger and G. H. Brown, Bur. of Stds., *Tech. Paper*, 7, p. 61.

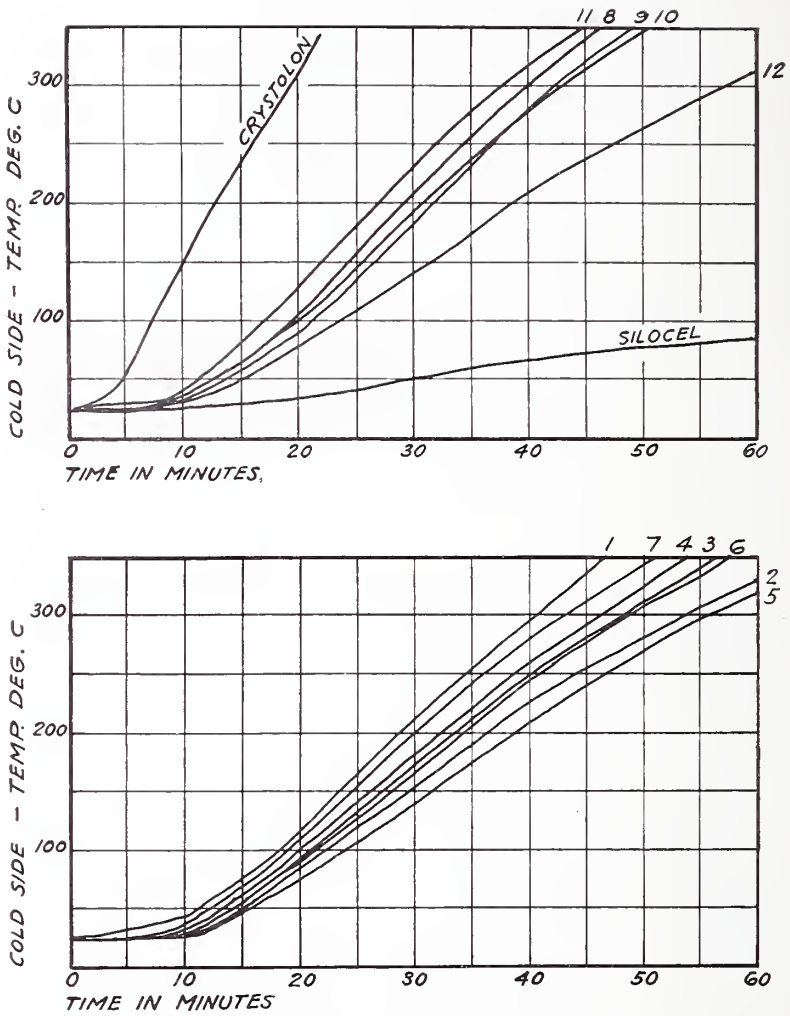


FIG. 3.—Relative insulation of $2\frac{1}{2}''$ brick. Temperature of hot side, 1100° C. ± 15° C.

but No. 3 (M87) and No. 7 (M94) deformed less than the poorest commercial brand. Four showed lower deformations than three of the five commercial bricks.

The *spalling test* results seem to bear no relation whatever to the bulk specific gravity, the porosity, the deformation under

load, or the cold strength. There is, however, quite a marked relation between spalling behavior and thermal insulation, as may be observed by reference to Figs. 2 and 3. This is especially interesting, notwithstanding the fact that the object of each of these tests was to obtain only comparative values for the different bricks. It presents an interesting topic for further study.

The character of the clays constituting the several bricks seems to have little influence on the spalling behavior. In the present study, however, the variety of clays used is too limited to warrant a general statement. The physical properties of the bricks seem to be the factors of real influence, though the manner in which each operates is not disclosed by the present set of data.

The *thermal insulation* data cannot be readily correlated with the compositions, for the field of compositions covered is too small. However, there are several interesting features to be pointed out. Contrary to our usual conception there is no relation, in the present case, between porosity and insulation property. In mixtures M91 and M94, the more porous brick (M94) is the poorer insulator. The same fact appears as well in M87, M88 and M89.

It should be noted that all of the experimental bricks are grouped within the extreme limits set by the commercial brands.

Conclusions.

1. The working properties and drying behavior of a fire clay mixture are affected by additions of sawdust in the same manner as by additions of grog.

2. Between 15 and 20 per cent of sawdust may be added to mixtures of the type studied without appreciably affecting the total shrinkage.

3. Raw kaolin increases the drying and burning shrinkages, but may be used without harm up to approximately one-third of the plastic clay content.

4. Not more than 45 per cent of sawdust can be added and still maintain a workable mixture. Forty per cent is about the practical limit.

5. At least 45 per cent of plastic clay (not more than 15 per cent kaolin) is necessary for proper working qualities.

6. The weight of the bricks is controlled largely by the amount of combustible material added, but the porosity and apparent specific gravity of the clay used has sufficient influence to be of importance.

7. In open-burning mixtures, additions of sawdust increase the porosity of the burned bricks by an amount equal to approximately one-half of the bulk volume added.

8. There appears to be some relation between the tendency to spall and the property of thermal insulation of the bricks tested in this study. The bricks of the better insulating qualities seem to spall the least.

9. There seems to be no relation between porosity and thermal insulation of these bricks, contrary to what would naturally be expected.

10. This study would indicate that approximately four pounds is the minimum weight that can be reached in a light-weight brick to satisfactorily compete against standard No. 1 fire brick and still retain sufficiently good working properties in the mixture to make commercial manufacture possible.

11. The load test appears to offer a better and surer means of judging these bricks than any other, for the light-weight bricks compare favorably with the commercial bricks in all tests except this.

12. In general the data presented offer some encouragement and indicate the possibility of producing, commercially, very satisfactory fire brick of very low weight.

Research Laboratories,
Norton Company
Worcester, Mass.

COMMUNICATED DISCUSSIONS.

D. W. Ross: In light-weight clay refractories for furnace linings, we must expect to sacrifice something in heat insulating value, for of necessity the weight of a brick depends largely upon its porosity.

This is in accordance with the results of the work of Ray and Kreisinger,[1] who found that in furnace walls fire bricks give

[1] W. T. Ray and Henry Kreisinger, U. S. Bur. Mines, *Bull.* 8 (1911).

greater insulation than a dead-air space, and who state that "when heat at low temperature is insulated, use air space; when the heat is at high temperature, as is the case in furnaces, use solids of poor conductivity."

In general, Kreisinger found that a 2-inch brick wall was more advantageous than a dead-air space above 352° C, and a 4-inch brick wall was more advantageous than a dead-air space above 227° C—when the measurements were based on a drop in temperature of 100° C. This in turn is in accordance with the laws of radiation and conduction. We know from the Stefan-Boltzmann law that radiation increases approximately as the fourth power of the difference in temperature between the hot and cold body, while the "amount of heat conducted through a unit of area from one part of a body to another is proportional to the temperature difference of the two parts; is proportional to the conductivity of the body; and is inversely proportional to the distance between the two parts of the body." Hence, as stated above, at furnace temperatures we would expect more space to decrease the insulating power of a brick to some extent, instead of increasing it.

With reference to the rate of cooling of clay fire-brick, above dull red heat, we know that silica and clay probably have slightly different thermal capacities and thermal conductivities, and that conductivity may be retarded in a brick by lack of contact of adjoining particles, due to porosity, or be otherwise affected by the degree of vitrification of the brick. We have a few data, however, that appear to indicate that the controlling factor in the cooling of clay fire-brick is the mass per unit of volume; that the rate of cooling (drop in temperature) decreases as the mass per unit volume increases, or, in this case, it is probably safe to say that in spite of differences in per cent of silica present, porosity and degree of vitrification, the controlling factor in the rate of cooling (drop in temperature) of a clay fire-brick, above dull red heat, is the actual quantity of heat stored in the brick.

Mr. Beecher's paper appears to the writer as an excellent contribution to the subject of light-weight clay refractories and is of special value because of the direct correlation of the light-weight

refractories with sufficient numbers of standard commercial brands of fire brick to give us a perspective of the relation of properties between the former and commercial fire brick.

At least where large percentages of kaolin have been used in the manufacture of light-weight refractories, the surface of the brick, where a gas or oil flame impinges directly upon it, soon shrinks and develops many cracks similar to those appearing on the surface of a sun-baked mud pond. Such high kaolin bricks, especially when very porous, are apt to contract (not necessarily soften and settle) in the load test. However, it is our opinion that with the right clays and methods of handling, a refractory brick of comparatively light weight can be produced that will meet gas and oil-fired furnace conditions and be well within the limits of the load test (1350° C and 50 pounds per square inch).

Laboratory
The Findlay Clay Pot Co.,
Washington, Pa.

R. L. Clare: Our interest in this class of refractories was caused by inquiries made by the Navy Department, both at the Brooklyn and Philadelphia Navy Yards, for a porous light-weight refractory having certain qualities. We submitted samples to the Philadelphia Navy Yard for testing and made the pieces for one installation from this type of material.

Ground cork (8–16 mesh) was used to secure the desired porosity. This was decided upon after numerous difficulties encountered in securing a uniform grade of sawdust. No two lots of sawdust were found alike, either in the kind of wood, or size and shape of the grains. Some of the grains would be more or less cubical, while others would be slivers. The cork as received eliminated all of these difficulties.

The balance of the mixture was made up of Florida kaolin, a plastic New Jersey fire clay and 4–16 mesh ground, fire-brick grog. Numerous mixtures were prepared, the most successful of which, when used for bricks only, was of the following proportions:

	Per cent.
8–16 mesh cork	49.2
Fire clay	15.7
Florida kaolin	22.2
4–16 mesh grog	12.9
	100.0

This mixture pugged easily, pressed well, and dried satisfactorily. It is very important, especially if shapes other than brick are made, that a safe drying mixture be obtained. The properties of this mixture when burned to Cone 14 were as follows:

Total shrinkage	10.3%
Absorption	50.0%
Bulk sp. gr.	1.1
Weight of $9'' \times 4\frac{1}{2}'' \times 2\frac{1}{2}''$ brick	4.0 pounds

This material should easily stand cone 31, although no softening point determinations were made. We know, however, that all the inorganic materials which entered into the mixture have softening points above cone 31. Load tests were not made, as no means were at hand within the time available, but such a test should be made. In comparing the properties of the above brick with those found by the author, it would seem that the porosity of this mixture is a little too high when used under load conditions under fire.

Apparently it is possible to add a higher percentage of ground cork than of sawdust to a mixture of this type, as we used 50 per cent cork and still maintained safe working properties, whereas the author maintains that only 40 per cent sawdust can be added safely.

In making slabs or other shapes larger than a regular brick, the warpage of the mixture needs serious consideration. The porosity and shrinkage must be materially reduced over that shown above, to be safe for shapes. The exact limits for pieces of stated sizes has not been determined.

FEDERAL TERRA COTTA CO.,
WOODBRIDGE, N. J.

R. M. Howe: It is hardly possible to offer more than a favorable criticism to the above paper. However, it is questionable whether or not such a heavy load should be applied to the light-weight bricks. Because of their nature the use of such bricks would tend to greatly reduce the load effect and, out of fairness to them, load tests using pressures proportional to the weight of the bricks might well be considered.

Some interesting data is at hand which concerns the more silicious light-weight bricks. A $9'' \times 2^1/_2'' \times 4^1/_2''$ brick is at hand which weighs 814 grams. Its fusion point is about cone 31. Under the tentative standard load test for silica brick, but with a pressure of 20 pounds per square inch, similar bricks sheared after being held one-half hour at 1500° C.

Mellon Institute,
Pittsburgh, Pa.

F. H. Riddle: The results obtained by Mr. Beecher in his work on light-weight refractories are very interesting, especially as there is a distinct demand for this kind of product for marine boiler installations. The data presented is very complete and checks the results obtained in our laboratory. The fact that the thermal conductivity did not vary with the porosity is probably to be ascribed to the size of the pores. When these are large, transfer of heat by radiation from one side of a cavity to the other becomes a factor which is less prominent than in the case of a large number of fine pores.

It might be interesting to add that the first government order for a product of this kind was filled by the Harbison-Walker Refractories Company at Pittsburgh. The work done so far seems to show that there is a distinct commercial application for light-weight refractories.

Bureau of Standards,
Pittsburgh, Pa.

M. F. Beecher: The writer does not entirely agree with Mr. Ross that we must expect to sacrifice heat insulating value for increased lightness in weight, when the latter is obtained by in-

duced porosity. The conclusions drawn by Ray and Kreisinger[1] do not cover precisely the insulation problem these bricks present. Carl Hering has discussed their work[2] and has pointed out that the phenomenon of "contact resistance" is probably involved as well as that of radiation, and therefore a furnace wall will offer better insulation if the customary air space is filled with sand or a similar loose granular material. Likewise, a single brick extending through a furnace wall will conduct more heat, and be hotter on the outer end, than an adjacent brick which is not continuous through the wall but has several intervening cracks or joints. Induced porosity may have a somewhat similar effect upon the transfer of heat, and for similar reasons. It does not necessarily follow that because a material is more porous it should be a poorer insulator. When we think of heat insulators generally, we usually visualize a light, porous material such as Silocel, Nonpareil brick, or cork. It appears, however, that the size and character of the cells or pores, as Mr. Riddle points out, may be the factor that governs the effect of induced porosity upon heat conductivity.

Mr. Clare's contribution to this discussion is very interesting and valuable. There are great differences in sawdust from different sources and care must be exercised if a uniform quality is always to be obtained.

The silicious light-weight brick mentioned by Mr. Howe, which weighs 814 grams, corresponding to a weight of approximately two pounds in the standard size, is very interesting. A more extended description would be an interesting contribution to the subject of light-weight refractories.

[1] Bur. Mines, *Bull.* 8 (1911).

[2] *Met. and Chem. Eng.*, 9, 438.

THE VOLATILIZATION OF IRON FROM OPTICAL GLASS POTS BY CHLORINE AT HIGH TEMPERATURES.

By J. C. Hostetter, H. S. Roberts, and J. B. Ferguson.

The presence of iron in optical glass arises from two main sources, namely, the raw materials and the pots used for melting.[1] Improvement in the purity of raw materials was established in advance of the development of good pots, with the result that for many months the amount of iron absorbed by the glass from the pot was several times that introduced from the batch. In the expectation of reducing the amount of iron contributed to the glass by the pots then available to an amount not greater than that introduced with the batch, the experiments described below on the volatilization of iron from pots by the action of chlorine were carried out. The experiments indicated beyond doubt that iron could be readily removed from pots by this method, which requires very simple equipment and can be applied during the "arching" or burning period; the cost of the process is negligibly small in comparison with other costs involved in making optical glass, and the technic required is simple. The large scale experiments to be described here were, however, only partially successful—not on account of deficiencies inherent in the method, for the iron was actually removed from the pot—but on account of other conditions. Inasmuch as there are many ceramic materials whose iron content can be decreased by this method, the details of this investigation are recorded here in order that our experience may be available to any others who are interested.

Iron Absorbed from Pots.—For illustrative purposes, Table 1 has been prepared in order to show the variation in the amounts of iron found in different optical glasses made in this country.

[1] Iron introduced into the batch during mixing is included here under that contributed by the raw materials; iron may also be introduced mechanically from the furnaces.

The amount contributed by the batch and also the amount absorbed from the pot are given.[1]

A considerable part of the iron introduced into the glass from the pot comes from the complete solution of a portion of the pot; that is, either the solution of a uniform layer from the inside surface, or the solution of pot "stones." Another part comes from selective action[2] of the batch or glass on the pot material; iron oxide, silica, and perhaps other oxides going into solution, while the remainder of the undissolved pot material crystallizes as sillimanite. The white line frequently seen between the glass and the darker portion of the pot material consists of a crystalline layer of sillimanite with interstitial spaces between the sillimanite fibers filled with glass. Such zones are usually less than a millimeter thick. Melts made in some pots showed also a zone of brown glass a few tenths of a millimeter thick in contact with the sillimanite layer; other similar pots of the same iron content did not show such a zone.

It must be emphasized that under some conditions a pot containing over 2 per cent iron oxide may actually contribute less iron to the glass than a pot containing less than 1 per cent. In other words, the physical condition of the pot plays an all-important role in resistance to pot corrosion. If a dense surface has been made on the interior of the pot and this has been thoroughly vitrified in the arching and burning processes the pot becomes exceedingly resistant and withstands the action of the batch;

[1] It should be noted here that the figures for iron introduced from the batch are higher than would result from materials now available. The experiments recorded here were all carried out before October, 1917, since which time considerable progress has been made in the development of better raw materials.

[2] Microscopical evidence on this selective action has been given by Bowen (*J. Am. Ceram. Soc.*, 1, 596 (1918)). The chemical evidence is simply that whereas the pot material may contain 10 or 15 times as much alumina as iron oxide, the glasses made in such pots show a much smaller ratio between these two oxides. Reference to the analyses made by Allen and Zies (*J. Am. Ceram. Soc.*, 1, 784 (1918)) of American and German optical glasses shows that the *maximum* ratio Fe_2O_3 : Al_2O_3 is 1 to 5.5 (with the exception of Jena O — 1209 the batch of which contains alumina). Our own analyses on other optical glasses show similar small ratios, as do also some unpublished determinations of our colleague, Dr. H. S. Washington.

under such conditions, the amount of iron absorbed from the pot may be reduced to 0.02 per cent or less in the glass.[1] On the other hand, pots containing smaller amounts of iron may introduce more iron into the glass because of poor condition of the interior—such as high porosity, cracks around grog particles, crazing, etc.—which encourages pot attack.

TABLE I.

Iron Absorbed from Pots by Optical Glasses.

Glass type.	Approx. Fe_2O_3 content of pot.	Fe_2O_3 from batch.	Fe_2O_3 in glass.	Fe_2O_3 from pot.
CROWN:				
Borosilicate.....	2.2 %	0.030%	0.080%	0.050%
Borosilicate.....	?	0.022	0.035	0.013
Light.....	3.0	0.025	0.074	0.049
Light.....	2.5	0.025	0.038	0.013
Light.....	0.5	0.025	0.031	0.006
Light Barium.....	2.2	0.027	0.053	0.026
Light Barium.....	2.2	0.027	0.075	0.048
Light Barium.....	0.75	0.035	0.043	0.008
FLINT:				
Baryta.....	0.75	0.025	0.035	0.010
Dense.....	2.2	0.017	0.056	0.039
Dense.....	0.75	0.017	0.020	0.003
Light.....	2.2	0.020	0.053	0.033
Light.....	2.2	0.020	0.037	0.017
Very dense.....	2.2	0.022	0.062	0.040

The weight percentage of iron introduced into the glass from the pot varies considerably, other conditions being equal, with the size of the pot and the density of the glass; the surface exposed to the glass increases as the square of the diameter while the capacity goes up as the cube. The weight percentage of iron is highest in the lighter glasses such as the borosilicate and light crowns and lowest in the very dense flints. These relations are made clear in Fig. 1, which gives the calculated percentage of

[1] The pot shown on the left in Fig. 5 contributed only 0.017% Fe_2O_3 to the light flint made in it. Note the sharp line of demarcation between pot and glass. The pot carried 2.2% iron oxide.

Fe_2O_3 which might be introduced into glass from the pot for different types of glass in pots of the usual sizes and proportions. The figures given here are based on the solution of all the iron from a uniform layer 1 mm. thick over the entire inner surface of the pot, and upon an iron oxide content of the pot taken as 2 per cent. Actually it has been found that pot corrosion is generally greater around the knuckle of the pot and across the bottom than around the sides. With dissolution of a millimeter layer in the 36-inch pot ordinarily used and an assumed iron content of 2 per cent Fe_2O_3, the percentage of iron absorbed from the pot

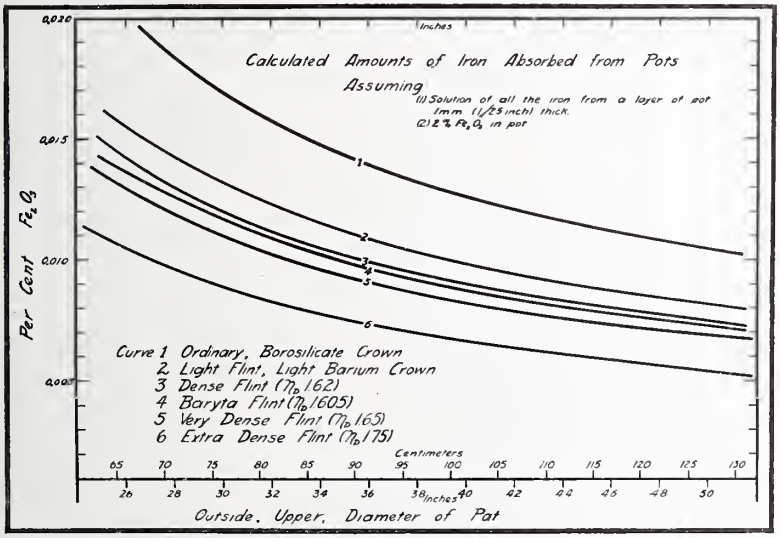


FIG. 1.—Calculated percentages of Fe_2O_3 in various glasses, derived from various sizes of pots, on the assumption of complete solution of the iron in a layer 1 mm. thick, and a ferric oxide content in the pot shell of 2.0 per cent.

is 0.014 per cent for a borosilicate or light crown and 0.010 per cent for a flint of index 1.62. The surface of the clay stirring tube exposed to the melt has not been considered in these calculations, although this surface is about 10 per cent of the inner surface of the pot. Under actual conditions the amounts of iron oxide absorbed from the pot are frequently several times those given in Fig. 1. (See Table 1.)

Use of Chlorides, or Chlorine, to Volatilize Iron.—The use of various chlorides for volatilizing iron from enamels is familiar, and has been described by Bole and Howe,[1] who found that from 8 to 36 per cent of the total iron could be removed from batches by this method. A method for the purification of sand has been described,[2] which is based upon the ignition of the sand with 2.5 per cent sodium chloride. The use of chlorine for the volatilization of certain metals is of frequent occurrence in analytical chemistry and similar reactions are taken advantage of in the purification of various compounds. Chlorine at high temperature has recently been used by Phillips to purify zirconia from iron.[3]

The possibility of volatilizing the iron from a pot before the pot was used was naturally suggested by the above-mentioned uses of chlorides and chlorine. After a few preliminary experiments, made on a small scale at the Geophysical Laboratory, had indicated the successful removal of iron by this method, one of the writers, in conversation with Mr. George C. Stone, of the New Jersey Zinc Co., learned that many years ago Mr. Stone had similarly used a stream of chlorine to bleach small clay crucibles.

Preliminary Experiments.

Recognizing the advantage to be gained by diminishing the iron content of the available pots if only by one-half, experiments were first made on a laboratory scale to find the best conditions for using chlorine and to learn how efficient it might be. These experiments were conducted by heating portions of previously burned pot shell, held in a porcelain or alundum boat placed in a silica glass (or, at the higher temperatures, a porcelain) tube through which the chlorine stream was forced. The tube was heated in an electric furnace and the temperatures, taken by a platinum platinum-rhodium thermoelement, were read on a millivoltmeter. The first experiments showed that chlorine at 800° C[4] would remove some portion of the iron but that better results were obtained at higher temperatures. The effect of

[1] *Trans. Am. Ceram. Soc.*, **17**, p. 125 (1915).

[2] J. G. A. Rhodin, Brit. Patent No. 8495 (1914).

[3] A. J. Phillips, *J. Am. Ceram. Soc.*, 1, 791 (1918).

[4] All temperatures in this paper are expressed in °C.

water vapor was later tested by passing the stream of chlorine through water before introducing it into the tube; the effect of hydrochloric acid gas was similarly tested; the results of these experiments will be given below.

Effect of Time and Temperature.—The details of several runs under different conditions are given in Table 2. From the results of these tests it is evident that the chlorine treatment should

TABLE 2.

Iron Removed from Pot Shell by Chlorine.
(Chlorine Flowing from Zero Time).

Minutes to reach maximum temperature.	Maximum temperature °C.	Minutes held at maximum temperature.	Iron content of pot shell % Fe_2O_3.		Percentage of total Fe_2O_3 removed by Cl.
			Initial.	Final.	
...	1100°	120[1]	2.19	1.03	53.0
155	1214	195	2.19	0.78	64.3
...	1225	105	2.19	1.20	45.2
185	1365	125	2.19	0.49	77.6

be conducted at the highest attainable temperature and should continue for at least 2 hours. The pot shell used here contained 2.19 per cent Fe_2O_3.[2] The residual iron, after a three-hour run at 1214°, is about 36 per cent of the total amount of Fe_2O_3 initially present, while after 2 hours at 1365° the residual iron is reduced to 22 per cent, showing conclusively that from $^2/_3$ to $^4/_5$ of the iron can be removed by this method.

The treated pot samples after these runs were nearly white in color with occasional yellowish spots scattered through the mass. The portions of pot were upwards of a centimeter in thickness and no difference in color was to be seen between the outer and interior portions showing that the gas had penetrated to the center. The appearance of the treated pot shell re-

[1] Approximate time.

[2] All results in this paper on iron contents of pots and glasses are calculated as per cent Fe_2O_3 without reference to the actual state of oxidation of the iron. Analyses by J. B. Ferguson, to be published later, show that glasses made in gas-fired furnaces may carry from 10 to about 30 per cent of the total iron in the ferrous state.

sembled very closely certain of the more refractory kaolin pots developed later. Some pieces of ordinary red brick were placed in the boat along with the pot shell in one run and after the treatment with chlorine they were beautifully white; the results of the chlorine treatment are evident from Fig. 2, which shows portions of treated and untreated brick. In another experiment an "Alundum" boat was used as the container for the pot shell; it also was bleached to a fine white, which did not show minute dark specks under the microscope as is frequently the case with alundum ware ignited in air.

Effect of Water Vapor.—Inasmuch as the products of combustion in a gas-fired furnace might interfere seriously with the volatilization of iron by this method it was desirable to investi-

FIG. 2.—Photograph showing treated and untreated ordinary red building brick.

gate the possible effects of such gases upon the reaction. Of the ordinary products of combustion, moisture might conceivably be the most objectionable since it would decompose the ferric chloride vapor with the formation of Fe_2O_3 and HCl.

The effect of water vapor on the reaction was studied by making a run with chlorine, which, previous to its introduction into the heated tube, was passed through warm water. A check run was made with dry chlorine. The results are given in Table 3. It is seen that whereas dry chlorine takes out 61.8 per cent of the

TABLE 3.

Effect of Moisture and of HCl on the Volatilization of Iron by Chlorine.

Chlorine.	Time at maximum temperature.	Temperature °C.	Iron content of pot shell, per cent Fe_2O_3.		Per cent of total Fe_2O_3 removed.
			Initial.	Final.	
Dry	30 min.	1265	1.94	0.74	61.8
Bubbled through water at 69°	30 min.	1265	1.94	0.88	54.7
Bubbled through conc. HCl at 17°	30 min.	1265	1.94	0.84	56.7

iron, chlorine carrying moisture, under identical conditions of time and temperature, takes out 54.7 per cent. These two figures are not so far apart as had been expected.

Effect of Hydrochloric Acid Vapor.—Prof. H. H. Willard, of the University of Michigan, suggested the possibility that hydrochloric acid might assist in the reaction and accordingly a run was made in which the chlorine was bubbled through concentrated hydrochloric acid before being introduced into the heated tube. The effect was here almost identical with that obtained with moist chlorine. The percentage of iron removed with chlorine and hydrochloric acid vapor was 56.7 per cent as against 61.8 per cent for pure chlorine and 54.7 per cent for chlorine and water vapor. There is no advantage to be gained therefore under these conditions from the use of hydrochloric acid vapor[1] along with chlorine. The details of the run with hydrochloric acid and chlorine are given in Table 3.

Large Scale Experiments.

The laboratory experiments having thoroughly demonstrated that pot shell could be freed of the greater portion of its iron by a simple treatment with chlorine at temperatures easily secured in glass furnaces the experimentation was next transferred to the glass plant of the Bausch and Lomb Optical Co., which very kindly

[1] Dry hydrochloric acid gas may be beneficial, its use, however, would require additional equipment (for generation and drying) which would not be readily adaptable to furnace hall operations.

placed equipment at our disposal. It is also fitting at this ti me to express our thanks to Mr. Victor Martin, glassmaker in charge, for the kind assistance which he rendered us.

These large scale experiments were carried out in order to learn the optimum conditions for volatilizing iron from glass pots by means of chlorine, and after these conditions were established, to chlorinate pots and study the quality of glass melted in pots so treated. The several runs will be described separately and full details given.

Necessary Equipment.—The equipment required for factory experiments was a source of chlorine (a cylinder of liquefied gas), a wash bottle containing concentrated sulfuric acid[1] connected to the cylinder to show the rate of flow of the gas, and a rubber tube connecting the wash bottle to a 15 mm. fused silica glass tube about 200 cms. long which served for the introduction of the gas into the heated pot. Furnace equipment of different types was at hand and some of the pots were treated in a recuperative furnace used for experimental work, others in pot arches, and one in a regenerative furnace. Temperatures were read with an optical pyrometer which was checked against thermoelements.

Treating a "Thread" Pot.—The first experiment on treating a "thread" pot was carried out in the recuperative furnace. In this test the pot was turned bottom up and chlorine was introduced beneath the pot. This test was qualitative only and no accurate record was kept of the amount of gas used, or the time of exposure. The temperature was kept at approximately 1200° and after treatment the pot was allowed to cool with the furnace. On examination next day it was found that the pot was noticeably bleached around the upper zones which, because of the relative position in the furnace, had been subjected to the most vigorous action of the gas. A deposit of beautiful hematite crystals was found around the edge of the pot where the ferric chloride vapors had come into contact with the furnace gases.

[1] Other liquids might have been used here but in choosing a liquid to show speed of gas it should be remembered that water vapor is to be avoided and sulfuric acid not only contributes no water but acts as a dehydrating agent.

The second experiment was also made with a "thread" pot covered somewhat as in Fig. 3A; the gas being introduced into the pot as shown in the figure. Chlorine was passed for two hours and again the temperature was around 1200°. The pot was cooled in the furnace as before and when cold was removed from the furnace and examined. In this case the pot was thoroughly bleached, as was also the fire clay slab used for a cover. Fig. 4 shows a photograph[1] of a section of the broken pot; the bleached zone may be readily seen. Deposits of hematite crystals were present around the upper portion of the pot and on the under side

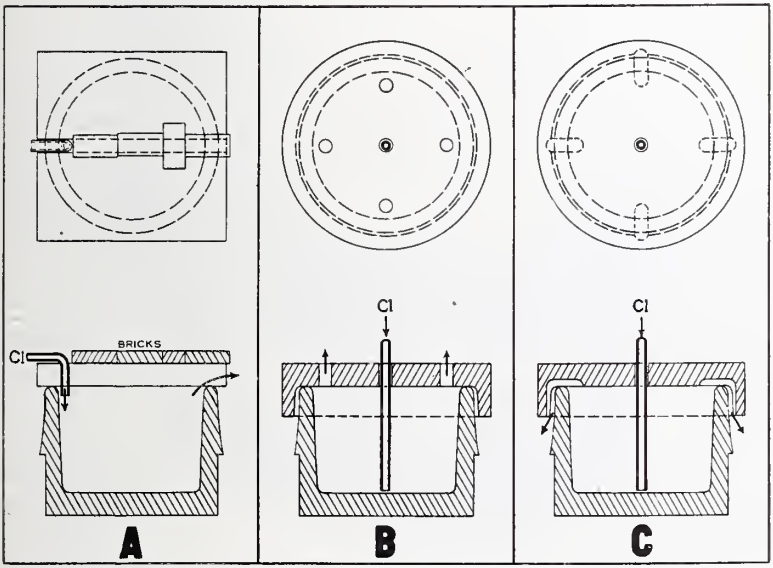


FIG. 3.—Plans and vertical cross-sections of pots, showing various methods of introducing chlorine.

of the cover where the effluent gases were met by the products of combustion in the furnace.

This test showed the practicability of the method as applied to large scale work.

Test on a Large Pot. Melt Number 573.—The next test was made on a type of pot used regularly at that time for the melting

[1] By Dr. R. H. Lombard, of the Geophysical Laboratory.

of optical glass and was carried out in one of the regenerative furnaces. The pot was covered in the manner shown in Fig. 3A and the chlorine stream was introduced through a silica glass tube bent as shown in the sketch. The pot was subjected to the action of chlorine for 2 hours at a temperature of 1225° when the fire-clay cover was removed and the furnace taken up to the filling temperature. When the cover was removed a cloud, which

FIG. 4.—Photograph of a section of a chlorinated pot showing depth of penetration.

probably consisted of hematite crystals, was seen to form within and about the pot and hence the results obtained later on the amount of iron extracted from the pot by the glass melt were rendered somewhat uncertain. The cover showed a beautiful deposit of hematite crystals where the chlorine and ferric chloride made their exit from the pot. Some of the crystals were 5 mm. on a side; they will be described in detail elsewhere.

A melting of dense flint was carried out in this pot immediately after the treatment with chlorine. Two other melts (Numbers

571 and 572) with similar (but untreated) pots and portions of the same batch were made at the same time in the other furnaces in order to have a check. The iron introduced into the glass from the batch was in all three cases 0.017 per cent Fe_2O_3. The glass from Melt 571 was later found to contain 0.056 per cent Fe_2O_3 and Melt 572 analyzed 0.055 per cent, showing that 0.038 per cent had been absorbed from the untreated pots. The iron content of Melt 573 was found to be 0.048 per cent Fe_2O_3, which means that the amount absorbed from the treated pot was 0.031 per cent. This difference, although less than had been expected, was nevertheless encouraging.

Experiment with a Large Pot in an Arch.—The experiment just described seemed to indicate that the chlorination had not been thoroughly effective for perhaps two reasons: first that since the chlorine had been introduced into the pot through a tube that did not extend much below the cover, the upper portions only of the pot had been freed from iron; and second that the precipitation of ferric oxide above mentioned had introduced some portion of these crystals into the pot where they were later dissolved by the glass. The next steps seemed, therefore, to be (1) to introduce the chlorine stream in such a manner that it first impinged upon the bottom of the pot, in order to remove iron where the greatest corrosion takes place during melting; and (2) to control the direction of the effluent gas so that precipitation of the ferric oxide formed by interaction of the ferric chloride with the furnace gases would not occur within the pot. Inasmuch as these conditions could not be readily met in the furnaces the next experiments were carried out in the arches used for drying and preheating the pots. The construction of these arches permitted greater latitude for manipulation than the furnaces, but on the other hand the maximum temperature attainable was considerably less.

The method of treating the next pot is indicated in Fig. 3B, which shows the entrance tube for the chlorine, reaching nearly to the bottom of the pot, and the method of covering. The pot used for this test was one which had been discarded because of cracks; it was of the usual 27-inch type. After the arch had been

heated to 1075° the pot was chlorinated for 2 hours at this temperature; the amount of chlorine used was 2.5 pounds. The pot was later taken out and examined. A deposit of hematite crystals had formed on the outer surface of the pot. The bleached portion of the pot was clearly evident in the cross section, and it was found that the bottom of the pot was bleached to a depth of about 15 mm., while the whitened zone became thinner towards the top. The result of this test was very satisfactory, therefore, in showing the best conditions for bleaching.

When the pot was taken into the sunlight, for purposes of photographing, the interior surface was seen to be covered with small glistening points. Examination under a hand lens showed them to be minute diamond-shaped crystals which were shown later by chemical and microscopical tests to be titanium dioxide. These crystals were studied in detail by Dr. H. E. Merwin and his results will be published in another place.

Chlorination in Arch. Melt Number 599.—The last experiment having demonstrated that the pot could be thoroughly bleached in those portions deemed most necessary, another pot was treated in an arch for 3 hours at 1007°, using the method of covering and of introducing the chlorine shown in Fig. 3B. Three pounds of chlorine were passed into the pot. Owing to accidental circumstances the pot stood in the arch four hours after it had been treated before being transferred to the furnace. Flames played over the pot during its treatment with chlorine and also during the period immediately before its transfer to the furnace.

A light flint batch was now melted in this pot, following the usual procedure. The batch contained iron equivalent to 0.02 per cent Fe_2O_3 in the glass. A proof taken during the fining process (while seeds were still present) showed 0.037 and the final glass[1] gave 0.048 per cent Fe_2O_3. Glass from the same type batch made in similar but untreated pots showed iron values from 0.053 to 0.058 per cent Fe_2O_3.

The bleached zone in contact with the glass is shown on the

[1] The absorption of light of Melt Number 599 was later found to be 1.4 per cent per cm. as determined by the Scientific Bureau of the Bausch and Lomb Optical Company.

right in Fig. 5; the brown glass layer, frequently observed in these pots, was absent.

The disappointing result obtained with this run was initially attributed to several factors. In the first place, the temperature

FIG. 5.—Photographs of cross-sections of pots containing melts, showing bleached (chlorinated) zone in the treated pot on the right. (Photo on left by Bausch & Lomb Optical Company; photo on right by Dr. F. E. Wright, Geophysical Laboratory.)

during chlorination was only 1000° and the preliminary experiments showed conclusively that considerably more iron would have been volatilized at a higher temperature. Much more serious appeared to be the fact that the pot, after the flow of gas had ceased, had stood filled with chlorine in the arch at the temperature of chlorination for four hours. During this period the

pot was surrounded with a reducing atmosphere which, penetrating to the interior of the pot, would tend to form ferrous chloride. The concentration of ferric chloride vapor would also increase, inasmuch as reaction between iron oxide and chlorine would continue at this temperature. When the cover of the pot was later removed—previous to the transfer to the furnace—a precipitation of ferric oxide would occur inside the pot and eventually this oxide would dissolve in the glass. The iron oxide precipitated under these conditions would probably not be wholly ferric but, under the reducing conditions obtaining here, it would contain ferrous oxide in solid solution.[1] The latter oxide would tend to produce a darker color in the glass.

Another possible cause suggested to us for the high amount of iron in the final glass was the presence in the pores of the pot of ferric chloride which was later absorbed by the glass. The following calculation shows, however, that such a condition would not increase the amount of iron in the glass to an unusual extent. The volume of pot walls and bottom may be taken roughly as 100 liters for the smaller size of pot used. Let us assume, in order to have the maximum value, that the pore space is 25 per cent. At 1000° the weight of ferric chloride vapor, assumed to be Fe_2Cl_6, occupying 25 liters, is about 90 grams, which is equivalent to 0.007 per cent Fe_2O_3 in the melt of average weight.

Large Pot Treated in Arch. Melt Number 610[2].—In order to avoid the possible difficulty caused by allowing an atmosphere of chlorine and ferric chloride to remain in the pot after treating with chlorine the next pot so treated was finally swept out with a stream of nitrogen from a cylinder of compressed gas. Fig. 3C shows the method of introducing the chlorine and also the method of covering the pot. In order to avoid the possible precipitation of hematite crystals where gases passed through the openings in the cover used previously (Fig. 3B), channels were cut in the under side of the cover, as shown in the figure, so that the precipitation would take place outside the pot. This pot was chlori-

[1] R. B. Sosman and J. C. Hostetter, "Solid Solution in the System Fe_2O_3–Fe_3O_4," *J. Am. Chem. Soc.*, **38**, 807–833, 1916.

[2] This test was carried out by our colleague, Dr. N. L. Bowen, to whom our thanks are due.

nated in an arch at 1075° for 5 hours; 7.5 pounds of chlorine were used at a uniform rate of flow. A light flint batch was afterwards melted in this pot but the result was not satisfactory. The melt contained many pot stones and there were other evidences of pot attack.

When the pot was later broken down the portions of the pot in contact with the glass were found to be thoroughly honeycombed, shelling off in layers and showing penetration by the glass to a depth of several millimeters.

Analysis of the glass from this melt showed the presence of 0.067 per cent Fe_2O_3, while the companion melt made as a check showed only 0.053 per cent. The inner layers of the pot were thoroughly bleached and this fact considered in connection with the high amount of iron found in the glass indicated that much more pot attack had taken place than normally. This was later confirmed by a determination of alumina in the glass from the treated pot and also from the check pot; the former carried 0.143 per cent Al_2O_3, while the latter contained only 0.067 per cent. This shows that at least 2 times as much pot dissolved from the shell of the treated pot—actually the amount of pot disintegrated was probably many times that from the untreated pot, because, as stated previously, the dissolving action of the melt is selective to a certain extent and relatively more iron than alumina is taken into the glass, a large proportion of the alumina persisting as pot stones made up of sillimanite crystals.

Cost of Chlorinating Pots.

At 20 cents a pound for chlorine the cost of volatilizing iron from a pot is of small moment. The maximum used in any chlorination (Melt No. 610) was 7.5 pounds at a cost of $1.50. The other necessary apparatus is cheap and the technic not difficult to carry out. If the process were adopted a permanent set-up could be made for each arch, or furnace, so that the chlorine could be readily connected and the entrance tube lowered into the pot. Hooded pots would present even less difficulty, as far as covering is concerned, than open pots.

Concluding Remarks.

The experiments recorded above point conclusively to the fact that iron can be volatilized from glass pots under factory conditions and that there is nothing difficult or impracticable in carrying out the process. The experiments also showed that the amount of iron in the pot could be readily reduced by half. The final tests showed less iron in the glass made in treated pots than in the glass made in untreated pots in all cases but one (the last test described above). The evidence all points toward the conclusion that the removal of iron from these pots—pots which depended in part upon iron as a bonding element—made them more porous and hence more readily attacked by the melted batch. Possibly these same pots would have been sufficiently dense even after chlorine treatment if a different burning schedule had been in operation at the time the experiments were made. The usual practice at that time was to set the pot in the furnace at 4 P. M. and to heat to 1400° by 7 or 8 o'clock. Later, a burning period of several hours at a higher temperature was added to the schedule, but no other experiments on volatilizing iron were made.

The discontinuance of these experiments, at a time when the many difficulties inherent in the early development of a new process bid fair to be completely solved, is regrettable, but was necessitated by the imperative demand for immediate maximum production of glass, rather than for the production of a glass of maximum transmission at a temporary sacrifice of capacity. Our experiences recorded here indicate that the process would function successfully with a pot in which the iron, though present, is not one of the bonding elements. Our later experiences in the arching and burning of pots make us confident that with proper heat treatment the disintegrating effect of the chlorine upon the pots in which iron is an essential bonding element could easily be overcome. In addition the application of this process to pot material, such as grog, would appear feasible, and this application alone would reduce the amount of iron in the final pot by one-third to one-half.

Geophysical Laboratory,
Carnegie Institution of Washington,
Washington D. C., April, 1919.

DISCUSSION OF AN ARTICLE BY G. W. MOREY ENTITLED "AN IMPROVED METHOD OF OPTICAL GLASS MANUFACTURE" IN THE FEBRUARY NUMBER OF THE JOURNAL OF THE AMERICAN CERAMIC SOCIETY.

By Clarence N. Fenner.

The article by Mr. Morey on "An Improved Method of Optical Glass Manufacture" in the February number of the Journal describes an interesting and valuable advance in the manufacture of optical glass. I think, however, that a word of caution may well be kept in mind. The inference from Mr. Morey's paper would seem to be that the process is applicable without restriction. That inference might at times lead one into difficulties. A circumstance which would be likely to render a longer period of fining advantageous or even essential would be the presence in the batch materials of those impurities, such as sulfates and chlorides, which tend to cause some glasses to become milky or opalescent. The presence of more than 0.30 per cent SO_3 or more than 2.0 per cent Cl in the potash used is likely to be dangerous (with some flints especially) and to make it desirable to hold the glass at an elevated temperature for several hours after melting is complete. In the early work of the men of the Geophysical Laboratory at the Bausch and Lomb works, milky glass was one of the chief evils with which they had to contend, and the amount of work which was devoted to finding out the cause of the phenomenon and learning what procedure was necessary in order to avoid it has left a strong impression with those who were engaged upon the optical glass work at that time, so that the possibility of the recurrence of such conditions is likely to be present in their minds. Later it was found possible to cut off the source of the trouble by requiring that the potash furnished by the manufacturers should meet definite specifications, based on the above-mentioned experience. It was only then, I think, when assurance could be had of a supply of pure raw materials, that shortening the fining process became feasible. It would be well for a manufacturer of optical glass, when considering the use of a twenty-four-hour schedule, to bear such limitations in mind.

Geophysical Laboratory,
Carnegie Institution of Washington,
Washington, D. C., April, 1919.

NOTES ON FIRE CLAYS OF THE NORTHERN APPALACHIAN COAL BASIN.

By Ellis Lovejoy, E.M., Columbus, Ohio.

The chief flint fire-clays in the northern Appalachian coal basin are:

(1) The Sciotoville in Kentucky. This is the Sharon horizon in Pennsylvania but in Ohio is known as the Sciotoville or No. 1.

(2) The Mercer in Pennsylvania and Maryland.

(3) The Lower Kittanning in Ohio (No. 5), Pennsylvania and Maryland.

(4) The Upper Freeport in Ohio (No. 7), Pennsylvania and Maryland.

The Brookville (No. 4 in Ohio) is reported to have excellent deposits of flint clay in Pennsylvania, and several other horizons are noted as bearing flint clay but none furnish any high grade material, or at least very little for the fire brick industries. The Brookville horizon has no flint clay in Ohio but it is an excellent plastic clay. In several localities in southern Ohio it has a peculiarity which has assisted the writer in working out some geological sections. In the localities mentioned, the outcrop does not tail down in the usual clay blossom but instead crumbles out in fine angular fragments and at a distance the waste resembles yellow sand, or more nearly sawdust. Close examination reveals the fact that each tiny fragment is conchoidal and in every visual respect a flint clay.

The Sciotoville clay is largely mined in Olive Hill, Kentucky and vicinity, and no where else so far as I know. It is a light-colored clay and in texture and fracture a typical No. 1 quality flint clay. This clay in relatively large areas has disseminated through it minute concretions of sulphide of iron, ranging in size from that of a pin head to microscopic grains. In some blocks the grains of pyrite are so small as to be practically invisible and their presence in detrimental quantity can only be determined by burning. This flint clay is found in pockets as far north as Logan, Ohio,

but in such pockets it is in the form of a boulder clay. The normal deposit has plastic clay immediately under the coal, underlaid by flint, and under the flint comes the semi-hard clay, but there is wide variation in the deposit. Not only is there variation in the thickness of each bed and in the quality of each clay but any one or two of the three may be entirely lacking.

The Mercer flint clay, sometimes designated as the Conglomerate clay in Pennsylvania because of its position relative to the Homewood and Connoquenessing sandstones is the most widely used and in general the best in the Appalachian basin. It is not found in Ohio, nor Kentucky, at least not in workable thickness, but it is extensively mined in central Pennsylvania and in the vicinity of Cumberland, Maryland. The bed cannot be said to have any typical structure. In the southern field it occurs in two benches with a division band of a few inches of sandstone or sandy clay. The benches vary up to fifteen or more feet in thickness with the flint clay in pockets embedded in the plastic. In central Pennsylvania the bed is normally thinner and is worked as low as three feet, including the plastic and "Burley" clay. The flint clay is more often found in the upper part of the bed, but so erratic is it in its position in the bed, that one is not justified in assigning it a normal position.

The Pennsylvania Geological Survey[1] sums it up as follows: "In the thick bed of plastic clay the flint clay may occur in any position in the same mine, sometimes at the bottom, sometimes at the top, or in any intermediate position, sometimes running out altogether, sometimes forming the whole bed, though as previously stated, there is almost invariably at least an inch or two of soft clay between the flint clay and an overlying coal bed, if the clay, as generally is the case, be overlain by coal."

I do not know the variations of the clay, but in so far as my observation goes, it frequently is widely different from a characteristic flint clay. It is harder than the other flint clays and some of it weathers down very slowly. In fact, some of it cannot be disintegrated by weathering within a practical period of time. Some portions of the bed lack the characteristic conchoidal frac-

[1] Topographic and Geologic Survey of Pennsylvania. p. 317, 1906–08.

ture and have not the dense smooth surfaces. Instead it breaks out in irregular shale like blocks with a granular surface. One familiar with flint clays would mistakenly pronounce this rough, apparently coarse grained Mercer flint to be of doubtful quality because of its appearance. This granular clay has a nodular structure to which the grainy appearance is due, and it is claimed that this rough clay is quite as refractory as the characteristically flinty clay from the same bed.

The Mercer flint clay is generally dark gray in color, which serves to distinguish it from the other flint clay horizons but the color is not to be taken as conclusive.

The nodular structure is peculiar and interesting. The nodules are not unlike bauxite concretions or "crystals" and I sometimes wonder if they are not genetically related.

In the Cretaceous kaolin deposits along the Fall line in Georgia, and similarly in Minnesota, are found extensive development of nodules or bauxite "crystals" and the only apparent difference between the true bauxite and the bauxite "blossoms," as the bauxitic kaolin is locally called, is that the latter is chalk white, while the true bauxite is gray in color. I have had analyses of the "blossom" nodules but they show kaolin only.

These nodular kaolins under cover are quite soft but have very little plasticity. They are readily cut out with an ax or saw and shaped up for building purposes and harden to a durable stone upon sufficient exposure.

The thought comes to me that the nodular kaolins, closely associated as they are in Georgia with bauxite deposits, have been subjected to the same conditions which developed the bauxite, except that the dissociation of the kaolin is only partial; that the silica set free by the dissociation has not been removed and to this soluble silica is due the subsequent hardening. An analysis naturally would show a kaolin ratio although the nodule might not be kaolin. I was told that the nodules in the flint clay in the Mercer horizon were higher in alumina than the normal clay which has practically a kaolin ratio, but an analysis which I made did not confirm this.

The Mercer clay beds which are worked in the Clearfield, Pa. district carry very pure flint clays and in my opinion rank first in

quality compared with any flint clays in the Appalachian basin. Farther east in the Lock Haven district the Mercer clay runs higher in iron but not to such an extent as to materially affect the quality of the product.

The Lower Kittanning flint clay is worked in Jackson county, Ohio, especially in the vicinity of Oak Hill, and extends north into Vinton county. Occasionally traces of it are found in central-eastern Ohio but it is not workable and is lost sight of in the vast quantity of excellent plastic clay at this horizon. Its best development in Ohio is in Tuscarawas county, northern Ohio, north of Canal Dover, around Mineral Point and particularly near Strasburg.

It is found in central Pennsylvania where it is largely used in the manufacture of fire bricks, but it is not the equal of the Lower Mercer clay and it is only used where the latter is not available. I have also found it in good thickness in Georges Creek, near Cumberland, Maryland.

It is not so hard and flinty as the Mercer or the Sciotoville and in color is neither a cream-buff nor a gray but instead an intermediate drab. Where mined near the outcrop it has a brownish color, probably due to extraneous iron, and in the mass a mottled-greenish cast.

The mottled color of this flint clay is evident in a number of localities. In Georges Creek above mentioned the flint clay is made up of a mixture of light and dark gray, or more nearly a matrix of dark gray clay in which are embedded angular masses of the light clay—all flint clay with no physical line of separation to account for or distinguish the two clays. It is my opinion that there is a difference in the two and that as the outcrop is approached the matrix is disintegrated to a soft clay, perhaps not plastic and in this event it would be the so-called semi-hard clay, in which remain the boulders of undisintegrated light-colored clay. The flint clay from this horizon in southern Ohio also has this variegated color, and thus we have this characteristic in two widely separated districts in Maryland and Ohio. I do not know whether the color peculiarity prevails in the Pennsylvania districts or not, but I am told that it does in some localities.

A question might be raised whether the Oak Hill clay should be identified as the Lower Kittanning, since it is found on top of the coal of that horizon, while the plastic clay for the manufacture of bricks in that locality comes from under the coal. The old theory that the coal grew on the clay bed precludes the possibility of the clay being above the coal. There are a number of sections which are pinched, in which one clay with its cover of coal rests immediately upon a lower coal with its underlying clay, and if the intermediate clay is thick we may identify the two coals as separate horizons, or if the intermediate clay is thin we may count the two coal seams as a split seam in one horizon, particularly if the horizons above and below are in their proper place. Where two horizons are thus near together the removal of the upper coal would leave one coal with a clay above it and below it.

Near Hamden, Ohio, there are twenty-five feet of plastic and sandy clay at the Lower Kittanning horizon. Upon this rests a foot or more of coal, then a foot to a foot and one-half of flint clay, similar to the Oak Hill clay, and this is covered by a foot of coal. The Middle Kittanning and Upper Freeport coals are in their proper places higher in the hill. Here then we have the Lower Kittanning coal bed in about four feet thickness with a flint clay seam between the upper and lower benches of coal. There is nothing unusual in this except that the clay seam is a flint clay. We have, therefore, the Lower Kittanning flint clay above the coal at Oak Hill, within the coal at Hamden, and under the coal in northern Ohio, Pennsylvania and Maryland, in so far as I have seen it.

Wilber Stout's theory[1] that the clay beds accompanying the coals are hydrated coal ash, whatever objection may be raised to it, will explain many perplexing problems—one of which is the question of clay seams in coal beds.

The Middle Kittanning, Pittsburg hand Pomeroy coals in Ohio, each of which in its full thickness includes a clay seam from six to twelve inches thick, are not exceptional in this respect. They also include slate seams. The latter we can explain by floods, however brought about, covering the peat bogs with mud

[1] *Tran. Am. Ceram. Soc.*, **17**, p. 557 (1915).

which subsequently hardened into stratified shale which in coal beds we call slate. By analogy the clay seam is a similar flood deposit but not so stratified and quite different chemically. The stratification is of no consequence because a series of floods would give the stratified shale while a single deeper flood would give the clay deposit, but originally the same material as the shale.

We explain that the coal bed took root in the clay seam and purified it, particularly in the removal of iron since the shale is red burning and the clay buff.

We cannot understand why the coal should have grown on one mud deposit and not in another and why a thin streak of coal between clay and shale, as often happens, should have purified the seam of clay where a heavy bed of top coal had no effect on the shale.

Stout's theory of oxidation and hydration accounts for the clay seams without disturbing the evident cause of the shale seams, and the clay beds may be under, within, or on top of the coal or without accompanying coal, depending upon the vagaries of the oxidation.

It is not my purpose to discuss Stout's theory, and I only mention it because it fits in with my contention that the Oak Hill flint clay on top of the Lower Kittanning coal is the Lower Kittanning clay, and it also serves, though not essential, in a question which I will bring up later.

The Upper Freeport flint clay is characterized by iron carbonate concretions en masse. Thus I have seen it in Bolivar, Pa., Georges Creek, Md., in less degree in southeastern Ohio, and the Pennsylvania Geological Survey[1] mentions the presence of "ore balls" throughout a relatively large area in that State. These concretions vary in size from that of a marble to that of a barrel and in some cuts, where weathering has exposed them by crumbling away the clay, they are the predominating mass of the bed.

This flint clay is found in southern Ohio where the silica content is usually higher than in the other flint clays. In a number of localities where I have examined it, good clay is found in the outcrop but under cover it quickly merges into a sandy material,

[1] *Loc. cit.*, p. 320.

oftentimes scarcely better than a coarse sandstone. It seems likely that these outcrops are merely remnant boundaries of pockets of clay; that the mass of the clay has been removed by erosion up to the more resisting sandy material. In several localities, notably Moxahaha and the Carbon Hill, Ohio, the remnant is of workable area.

In recapitulation, in so far as my observation goes, the general

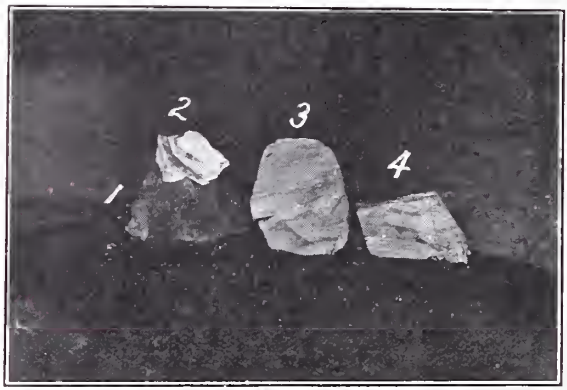


FIG. 1.—Specimens of Upper Freeport flint clay from the same mine.

No. 1. Blood-red flint clay.
No. 2. Ditto, burned to a buff color.
No. 3. Gray flint clay flamed with streaks of red, like "Aurora Borealis."
No. 4. Gray flint clay, veined.

characteristics of the four clays are as follows:

Sciotoville clay has a light color, close structure, conchoidal fracture and contains minute disseminated pyrite concretions.

Mercer clay is dark gray in color, exceptionally hard, weathers slowly, its more or less nodular structure giving it a granular appearance and this granular clay has not conchoidal fracture. The dense smooth portion of the clay is characteristically conchoidal.

Lower Kittanning clay is usually light, but frequently decidedly mottled in color; not so hard as either of the above nor so sharply conchoidal in its fracture; averages higher in silica,

which is frequently in the form of quartz grains embedded in the clay.

The Upper Freeport clay is light in color; contains accompanying iron carbonate concretions and frequently merges into very coarse sandy clay.

Physical Differences in Fire Clays.

It seems to me that Stout's theory above mentioned gives us a better starting point in an effort to explain the physical differences of plastic, "semi-hard," and flint clays.

Kaolins developed *in situ* are as a rule only slightly plastic, while alluvial kaolins and clays possess, in variable degree, greater plasticity and I think it cannot be controverted that the primary plasticity which these alluvia possess is due to the attrition which they receive during transportation. I use the word "primary" to designate the plasticity which a clay has when it is deposited and to distinguish it from any plastic development subsequent to such deposition. I am of the opinion that clays undergo changes in their plasticity after deposition. We know, of course, that plasticity is destroyed by heat and likely in some measure by pressure but, as I believe, we do not know that plasticity is increased following deposition.

All the shales of the coal measures are more or less plastic and while there are marked differences in the degree of plasticity, which cannot be fully accounted for by mineralogical differences, yet we get no such plasticity differences in the shales as we find in the fire clays, especially considering the close relation of the flint, "semi-hard," and plastic clays in each bed. The clays are practically chemically alike and it is inconceivable that the marked physical difference so sharply defined can be attributed to a primary cause; in other words, that these clays, if alluvial, came to their final resting place with the differences they now show.

It may be true, in fact it likely is true, that the pressure which has hardened the clays and shales, together with some heat development, has reduced the primary plasticity but we cannot ascribe to these causes the marked differences in contiguous flint and plastic clays.

Bacterial action has been frequently mentioned as effective in

developing colloidal conditions to which plasticity is ascribed and the presence of algae in clay has been given in explanation of the deep red color which some clays have. I have seen red fire clays which burned buff and the red color of the clay could not be due to iron, but instead was said to be in consequence of the presence of red algae. Notable examples of such red fire clay are found in the Upper Freeport horizon in southeastern Ohio. Numerous outcrops with a flaming red color are seen, and we also get pockets of red clay in the solid mass under cover, a thousand feet from the outcrop. All this red clay burns to a buff color and is quite refractory. Another peculiarity of flint clay in this locality is a marked venation, often broken and distorted, which seems to me not to be due to stratification.

The deep-red clays of Nova Scotia burn red but the burned color is not as deep a red as that of the raw clay, and this is also true of a deposit in Delaware which the writer developed several years ago. Whether the red color of these raw clays is in part due to algae, or not, I do not know, but in both localities the red clay is accompanied by muck clays favorable to the growth of bacteria or algae. In both instances these clays are extremely plastic. In boring into them with an auger, and in drawing the auger, the spiral of clay will frequently slip from the auger and the latter comes to the surface swept clean. When the auger holds its load of clay the latter streams out from the point of the auger in an attenuated string and may be loosened and slipped from the auger in a full length curl.

Some of the very red clays in the south burn a pale red, less deep in color than the raw clay. I am aware that these color differences in the raw and burned clay may be accounted for by iron solutions and agglomerations, except perhaps in the case of the fire clay mentioned, but the presence of algae offers a simple explanation.

In several instances I have found intimate irregular, I may say angular, mixtures of red and gray, or greenish gray clays. A notable example is in a clay mine about two miles north of Charleston, W. Va. The clay is fifteen to twenty feet thick and throughout it is an intimate mixture of red and greenish gray clay without any physical lines of demarcation between the two colors. In

the bottom of the bed is a fragmentary streak of flint clay. It was being mined under a solid roof without a trace of leakage and the mine was dusty throughout. It is inconceivable that the color difference extending to all parts of the bed without preponderance of either color in any part could be due to the oxidation of iron in the red patches. I am of the opinion that the color phenomenon of this bed is due to algae or other vegetable growth. Another instance is in southern Ohio where the peculiar appearance led the owner to some development work in search of precious metals.

If bacteria will develop in clay mixtures in storage cellars and increase the plasticity, as some potters claim, similar development will occur in clay bogs, and such bacteria, algae and other low forms of plant growths undoubtedly do occur in bogs whatever their effect on the clay mass. If a short period in a clay-storage cellar will materially increase the plasticity of a clay, we may expect a pronounced effect in the prolonged period of a geological accumulation.

Stout's theory of fire clays gives us a bog material primarily possessing little or no plasticity. It also explains the chemical differences of fire clay and shales, notwithstanding their juxtaposition. In these two factors it is favorable to this discussion, but not necessarily essential. We may assume that the fire clays and shales are transported materials; that the chemical differences are in consequence of the sources from which the materials were derived; that they possessed some primary plasticity, variable because of the variable material and the conditions of transportation; and finally that this plasticity was partially or completely destroyed by subsequent pressure with some heat development, or in other words, that the plasticity is destroyed to the extent that the material possessing little primary plasticity became nonplastic, while the more plastic material simply became less plastic.

With such a material in a bog teeming with protoplasmic growth, it is readily conceivable that there would be an extensive development of colloids, from the low forms of plant and animal life perhaps, and a colloidal condition in the clay material in consequence of the attrition of the grains through protoplasmic pressure and movement continued during a long period.

Protoplasmic growth is very sensitive to conditions and in the great area of plastic fire clays it is also conceivable that some small portion would be too pure to supply essential protoplasmic food, or that, because of acid, alkaline, or other chemical conditions, the protoplasmic growth would be retarded or prevented; that bordering this material there should be clays only feebly responsive to such cellular growth; and finally that, outside the influence of these unfavorable plots, the growth could be normal or excessive as conditions would permit.

It is not necessary to assume that there were no spores in any portion of the bed, but instead that some portions of the bed were unfavorable to their development and in consequence the colonization was slight. We would have, therefore, beds of light-burning clays with little initial plasticity. In some small portions there was no colloidal development and consequently no increase in plasticity; in other small portions colloidal development slightly increased the plasticity; in the great mass, however, over wide areas, the conditions would be favorable for protoplasmic growth with consequent colloids and resulting plasticity.

By subsequent pressure the bed is hardened into a rock and likely some measure of its plasticity destroyed, and this rock we are now mining and by grinding and pugging developing its latent plasticity—none in the flint, slight in the "semi-hard," and variably excellent in the plastic.

S. L. Galpin[1] presents a theory in explanation of the physical differences in fire clays diametrically the reverse of the writer's suggestion in the above notes. Galpin accepts the theory of sedimentary deposits and their purification by plant life, with the expressed opinion that these sediments were "largely colloidal." He starts with an exceedingly plastic mass whereas the writer's contention is that the mass is likely low in plasticity.

From this plastic material Galpin explains, that the flint clays are developed by setting and recrystallization of the colloidal sediments; that the "semi-flints" are derived from the flint clay through metamorphism by pressure and heat resulting in mineralogical changes; that the plastic clay associated with flint clay is

[1] S. L. Galpin, *Trans. Am. Ceram. Soc.*, 14, 336–345 (1912).

weathered "semi-flint" clay and it is structurally and mineralogically different from other plastic fire clays which have never been "set."

COMMUNICATED DISCUSSIONS.

R. R. Hice: A review of the fire clays of the northern Appalachian coal basin is certainly most timely, and especially so in view of the enormous demands which have been made upon them, and the constant call for better and ever better fire-resisting materials.

In Pennsylvania, the most of the flint clays are produced from the Upper Mercer horizon. I think the variations in the Mercer clays have been well brought out by Mr. Lovejoy, and while it must be said some of these clays do not have the usual flint clay appearance, yet that fact does not take away their value as refractory materials, but is simply another complication to be taken into consideration in any satisfactory theory of the origin of flint clays.

I do not think the Lower Kittanning clay is worked in Central Pennsylvania to the extent indicated by Mr. Lovejoy. In Clearfield and Clinton Counties, for example, it is the Middle and not the Lower Kittanning clay which is used. In the clays underlying the three Kittanning coals, the occurrence of flint clay in the Lower Kittanning horizon is quite unusual. The promising flint clay horizons of the Allegheny series are the Middle Kittanning and the Bolivar. The Lower Kittanning is quite often used for bonding material, often being shipped quite a distance for that purpose. At one locality there is considerable flint clay in evidence but it is scattered throughout the bed in small nodules, seldom as much as one-half inch in diameter.

It is an obvious error to correlate the flint clay of Bolivar with the Upper Freeport horizon. The two beds are distinct and separate, not only at the type locality, but at other points as well. Forty years ago Stevenson, in describing the section at Bolivar, distinctly pointed out the fact that the non-plastic Bolivar fire clay did not underlie the Upper Freeport coal, but was separated from it by several distinct strata. It has been said by some that the horizon of the Bolivar clay is the same as that of the

Upper Freeport limestone, and that the flint clay, when present, replaces the limestone. It may be this is often the case, but there are exposures which distinctly show both the fire clay and the limestone present. There has been considerable confusion caused by this wrongly calling the Bolivar the Upper Freeport. It is seldom when the flint clay is present that the true Upper Freeport is not found.

I must object to the loose way in which the term "kaolin" is used. If the term "kaolin" is to be used to designate any white clay, then let us use the term "white clay" and we will know what is meant. Dr. Sellards is to be congratulated that in his last Florida report, in speaking of the white clays there, he designates them as "ball-clays," which is really what they are, and the same term should be used to designate a great many other alluvial white clays. The word "kaolin" is rapidly coming to have no fixed meaning in the ceramic industry, being daily exploited by clay producers for purely commercial purposes, where they have materials that no one would pretend to substitute in a batch for kaolin. Neither should the term "kaolin" be applied to *all residual* clays, many of which are white but which do not possess the character and qualities of kaolins, and are not formed from the decomposition of igneous materials, but from secondary rocks.

As fine grinding and washing of a true kaolin increase the plasticity of the resultant remaining material, and we still call the remaining material kaolin, it has been suggested we should call the clay material which is broken down under natural conditions and been washed by nature and not artificially, a kaolin also. The very facts are an argument against this use of the term. Very fine grinding, accompanied by washing, is artificially producing the same conditions, but not in the intensified form that nature has used in the formation of sedimentary clays from once igneous rocks. As a matter of fact the extremely fine artificially ground and washed material should not and cannot properly be called "kaolin" without a modifying adjective. If the term "kaolin" is to be applied to plastic or semi-plastic white or varicolored clays formed by sedimentary deposition of material derived from the decomposition of igneous rocks at a considerable distance, being deposited and broken up and redeposited again

a number of times, we should extend the use of the term to embrace any clay whatever, and a common ordinary red-burning brick clay, found along a stream, or a hard laminated shale, should also be termed "kaolins," because they have been deposited in exactly the same manner, and it is possible to show the many steps in the transformation of material directly derived from the decomposed igneous rocks, through semi-plastic and plastic white clays, into various colored clays, with changes in the impurities present, into and through all lower grade materials, until finally deposited as mud banks along the ocean shore, and no one can draw the line when it ceases to be "kaolin," except where it leaves the parent bed—when it first changes from a residual material into a transported one.

State Geologist, Pennsylvania,
Beaver, Pa.

H. Ries: The occurrence of nodular bodies in many refractory clays, which has been touched on by Mr. Lovejoy, is one that has been described by different observers, but the origin of which has never been explained for all. We are, of course, very familiar with this structure in bauxite, where it is almost universally found, but it is not so common in refractory clays.

Mr. Lovejoy refers to it in the white sedimentary clays of the Lower Cretaceous in Georgia. Here we find lenses of white clay in the Lower Cretaceous, which are sometimes associated with true bauxite,[1] but within the clay itself there are sometimes lenses of nodular material, which are not bauxite, but consist of a mixture of kaolinite grains and quartz.[2] Nodular bodies have also been found in some of the Pennsylvania fire clays, but so far as I know they have not been examined petrographically to determine their mineral composition.

Some years ago Mr. Greaves-Walker[3] described a peculiar flint clay from Kentucky which contained similar oölites, which

[1] H. K. Shearer, A Report on the Bauxite and Fullers Earth of the Coastal Plain of Georgia, Ga. Geol. Surv., *Bull.* 31 (1917).

[2] R. E. Somers, Microscopic Examination of Clays, *J. Wash. Acad. Sci.*, 9, No. 5, 113 (1919).

[3] A. F. Greaves-Walker, "Note on a High Alumina Flint Clay," *Trans. Am. Ceram. Soc.*, 9, 461 (1907).

were subsequently found to be made up of the mineral gibbsite.[1] Mr. Walker, I believe, suggested the name "aluminite" for this clay, but as this has already been applied to a mineral it would have no validity.

A curious example of nodular clays is that found in the flint clay district of Missouri, where the material, sometimes made up entirely of small round bodies, occurs as irregular masses in the flint-clay deposits. In this case the nodules are composed almost exclusively of the mineral diaspore.[2]

I cannot agree with Mr. Lovejoy in his use of the term "kaolin." This was originally applied to white residual clays, and its application to sedimentary ones has in my opinion been the result mainly of commercial propaganda. It should be restricted to its original use. The two classes of white clay are not alike in their characters, nor can we even say that they are used for the same purposes, except perhaps in a very few cases. A somewhat similar confusion exists regarding the terms kaolin and kaolinite. The former is a rock name, and does not represent a substance of definite chemical composition, consequently there cannot be a kaolin ratio as Mr. Lovejoy states. Kaolinite, on the other hand, is a mineral of definite composition represented by the formula, $Al_2O_3.2SiO_2.2H_2O$, and may occur in almost any kind of clay, although it is most abundant in the high-grade clays, although even here it may not be as abundant as mica.[2]

CORNELL UNIVERSITY,
ITHACA, N. Y.

H. A. WHEELER: Mr. Lovejoy's generalizations that shales are always plastic but to variable degrees, while the fire clays range from marked plasticity to non-plastic in the case of flint clays, also holds good in the Missouri deposits.

The Missouri flint clays, which occupy small, local, disconnected basins in sink holes in pre-Pennsylvanian limestones (Mississippian and Silurian) usually weather in fine, angular granules on the outcrop, but in a few instances they become plastic for a few feet from the surface.

[1] S. L. Galpin, *Trans. Am. Ceram. Soc.*, **14**, 301 (1912).
[2] R. E. Somers, *Loc. cit.*

That the coloring matter in some red and pink clays is organic and hence burns out is true of a few Missouri clays, as observed by Mr. Lovejoy. In some instances, I think this reddish coloring matter will be found due to petroleum, where the oil has gradually infiltrated through the clay, in doing which more or less refining and refractionating occurs.

408 Locust St.,
St. Louis, Mo.

Ellis Lovejoy: I have examined a number of the flint fire clay deposits in Pennsylvania but chiefly the Mercer clays, and I had very little first-hand information in regard to the Lower Kittanning flint, and so stated. I am glad to be corrected by Mr. Hice but I get some comfort from the report of the Topographic and Geologic Survey of Pennsylvania, 1906–1908, page 312, wherein I find under the heading of Lower Kittanning Clay, the statement: "In many parts of the area a valuable flint clay is associated with the plastic clay." And again on page 320 is the following: "Mention has already been made of the wide extent of flint clay associated with the plastic clay under the Lower Kittanning coal."

Relative to the Upper Freeport coal, in the Fourth Series, *Bull.* **20,** Ohio Geological Survey, is the statement that the clay is directly below the coal, and also mention is made of the concretionary matter in the clay. I do not recall ever finding the coal and clay together in the same opening in Ohio but I have found them on opposite sides of the same hill and at the same level so far as it was possible to determine. In a mine in George's Creek, Md., cutting through all the coals from Brookville to the Upper Freeport inclusive, the coal of the latter horizon rests immediately upon the clay. I am pleased to have Mr. Hice bring out the fact that the clay in Pennsylvania does not underlie the coal.

The clay, whether with the coal or separate from it, is concretionary in the several districts mentioned. Petrographically, it is alike wherever found throughout the basin, and widely different from any other clay in the same area.

The geological conditions under which the bed was developed

were the same and have given the clay a marked feature in the unusual number and size of iron carbonate concretions.

The character is so marked as to correlate the clay as the same horizon in the three states. Over the wide area represented, it would not be strange if some portions of the bed of the clay had been covered with sediments upon which the coal growth again took root, without any break in the continuity of the coal bed. Splits in coal beds are frequently found and why may we not have a split by wedged shaped masses of sediments between a coal and a clay without breaking the continuity of either the coal bed or the clay?

If the coal accompanying the clay in Ohio and Maryland is the Upper Freeport, then in my opinion the clay in Pennsylvania belongs to the Upper Freeport horizon.

I do not wish to defend my use of the term "kaolin" ratio. It was loosely used but the average clayworker understands kaolin to mean a white clay with approximately the composition of kaolinite.

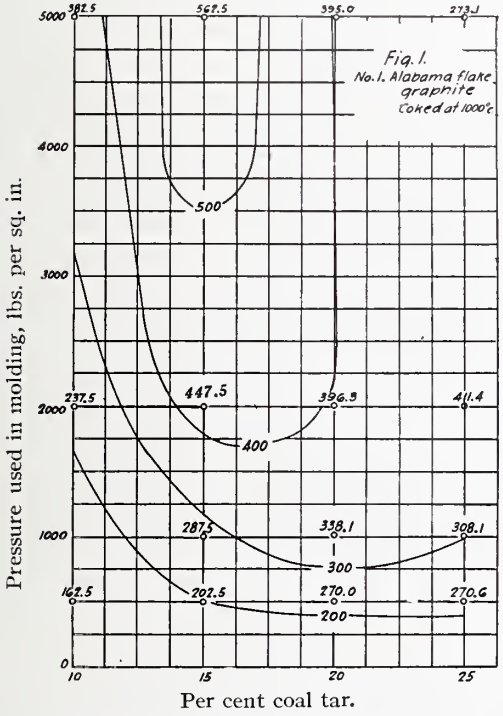
The nodules from the Georgia deposit could not have been a mixture of kaolinite and quartz, as suggested by Dr. Ries, because the silica content of such a mixture would be higher than that of kaolinite, or "kaolin ratio," as I put it.

I am of the opinion that the nodule in question was in a transition stage from kaolinite to bauxite, with none of the resulting minerals removed. Analytically, it had the composition of kaolinite, but mineralogically it was likely a mixture of a highly aluminous mineral—perhaps bauxite—and quartz, with or without undecomposed kaolinite.

EFFECT OF VARIABLE PRESSURE AND TAR CONTENT ON THE BRIQUETTING OF ALABAMA GRAPHITE.[1]

By R. T. Stull and H. G. Schurecht.

The Ceylon graphite occurs in comparatively large lumps and when ground and screened produces a material having a granular



Effect of tar and pressure on compressive strength of coked Alabama graphite.

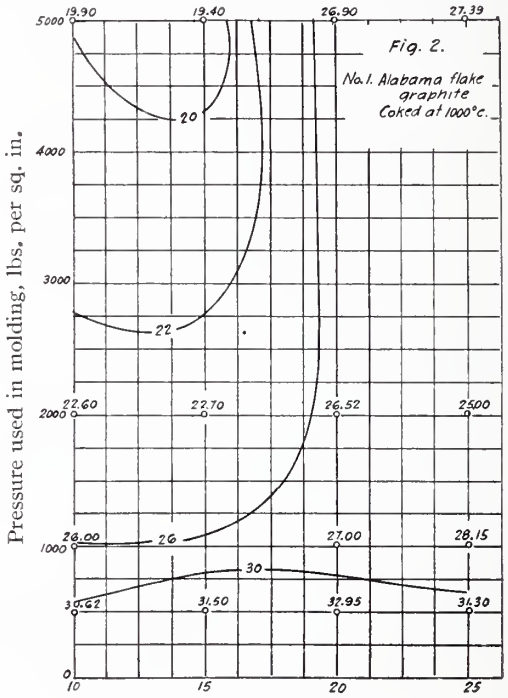
or splintery structure. On account of the comparatively large size of the lumps, it can be ground to almost any size of meshing desired. On the other hand, the Alabama graphite when pre-

[1] By permission of the Director, U. S. Bureau of Mines.

pared for the market occurs in a thin flat plate-like form seldom coarser than a 20-mesh size.

The argument which has been advanced by crucible-makers—to support their contention that the Ceylon graphite is superior to the flake form for the manufacture of crucibles—is that the granular and splintery graphite grains produce a stronger and

The effect of tar and pressure on the porosity of coked graphite.

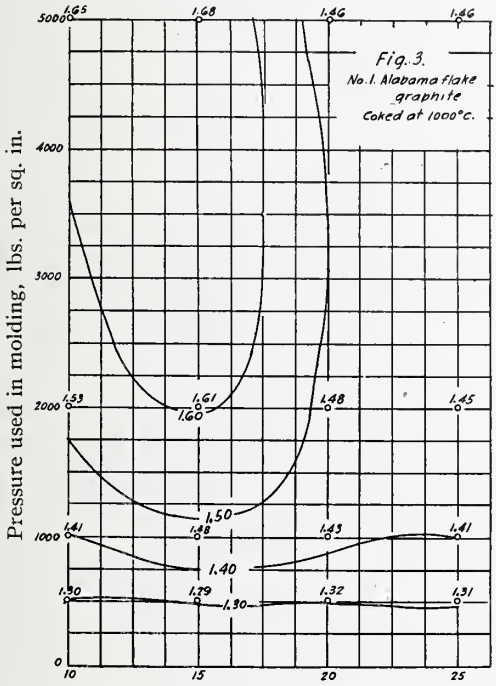


more durable crucible, due to the interlocking or "tying in" of the granules and splinters with the bond clay. On the other hand, the structure in the flake graphite body appears to be that of over-lapping plates which, it is claimed by a minority of domestic crucible makers, when bound together produce a stronger body than the granular form.

It was therefore suggested that if the Alabama flake graphite

could be modified in such a manner as to produce a granular form, larger than 20-mesh in size, the crucible making properties of graphite so prepared could be compared with those of the flake and the Ceylon, thereby throwing some light upon the question. Experiments were made to convert the Alabama flake graphite to the granular form by briquetting the graphite with

Effect of tar and pressure on the density of coked Alabama graphite.



tar, coking, crushing and screening. In order to produce a high degree of "compactness" and to approximate the Ceylon grain as closely as possible, a study of the effects of different briquetting pressures and different per cents of tar binder was undertaken.

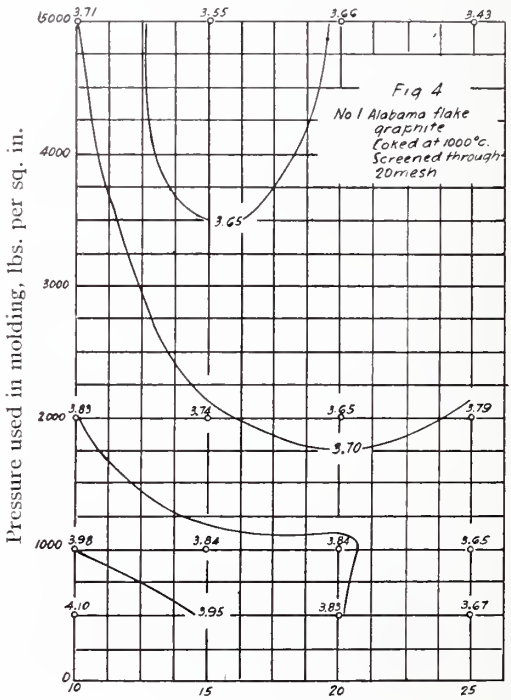
Two series were made. In Series 1 the Alabama flake graphite, screened between 20- and 100-mesh, was mixed with 10, 15, 20 and 25 percent coal tar (by weight), briquetted at different pres-

sures up to 5000 pounds per square inch on an Olsen compression testing machine, and coked rapidly at 1000° C.

Series 2 was a duplicate of Series 1 except that a mixture of three parts flake graphite and one part graphite dust passing the 100-mesh screen was used.

In order to determine the best pressure and per cent of tar to

The effect of tar and pressure on the bulkiness of Alabama coked graphite.



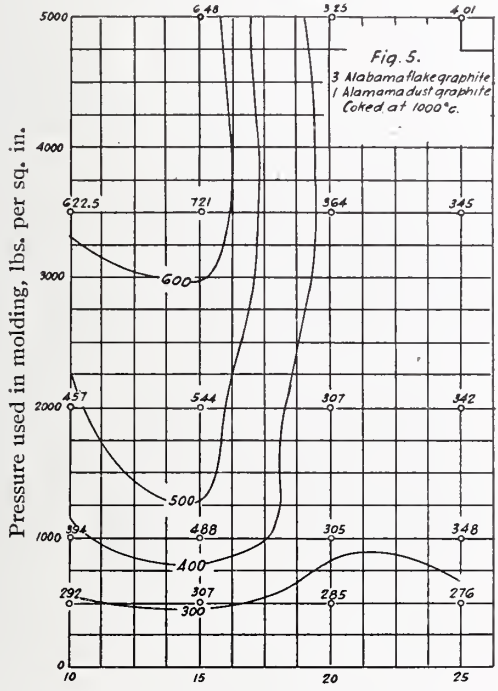
employ, the crushing strength, porosity, apparent density and bulkiness measurements were made on the coked samples.

The compressive strength tests were made on five check trials for each different set of conditions on cylinders $2^1/_2''$ in diameter and $3''$ high.

The porosity and apparent density determinations were made by the kerosene oil-immersion method on three check trials.

Bulkiness measurements were made by crushing the coked briquettes and screening between 20 and 100 mesh in order to secure results comparable with the tests on the 20-mesh flake graphite. Eighty grams of the prepared material with about 140 cc. of distilled water were placed in a graduated cylinder and the graphite allowed to settle for 24 hours, when the height

Effect of tar and pressure on compression strength of coked Ala. graphite.



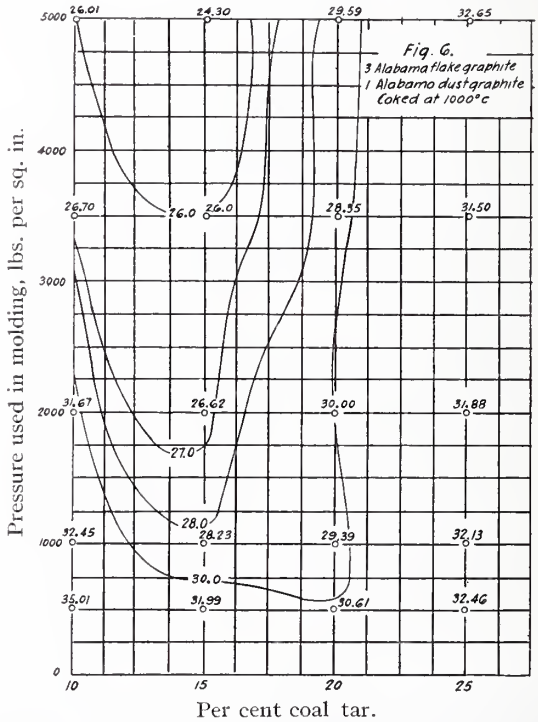
of the graphite in cubic centimeters was noted. The bulkiness was expressed in terms of the ratio of the number of cubic centimeters of graphite observed, divided by its true volume. For comparison, bulkiness measurements were also made on Ceylon, Canadian, and No. 1 Alabama flake graphites. All were screened between 20 and 100 mesh.

The comparative bulkiness was found to be as follows:

	Bulkiness factor.
Ceylon graphite	2.36
Canadian graphite	3.51
Alabama coked	3.54
Alabama flake	3.90

In order to show graphically the results obtained, crushing strength, porosity, apparent density and bulkiness lines were

Effect of tar and pressure on porosity of coked Ala. graphite.

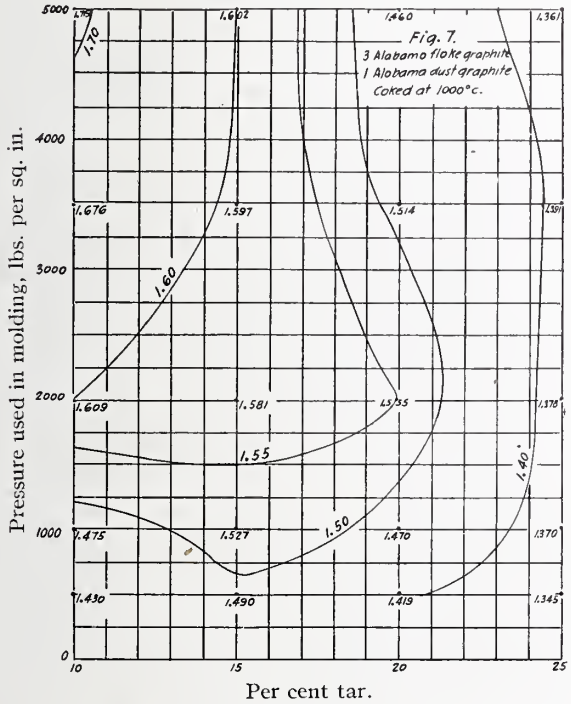


plotted. The pounds per square inch under which the briquettes were compressed are spaced on the ordinates and the percentages of tar spaced on the abscissae. Figs. 1, 2 3 and 4 show the results for Series 1, and Figs. 5, 6, 7 and 8 for Series 2.

As anticipated, the curves show that highest crushing strength, lowest porosity, highest density and lowest bulkiness occur

with highest pressures of briquetting in both series. In Series 1 for the flake graphite, the curves show that about 14 to 15 per cent tar gives the maximum crushing strength, minimum porosity, maximum density and minimum bulkiness for the highest briquetting pressures, and, in general, as the briquetting pressure decreases the amount of tar must increase up to a limit of ap-

Effect of tar and pressure on the density of coked Ala. graphite.



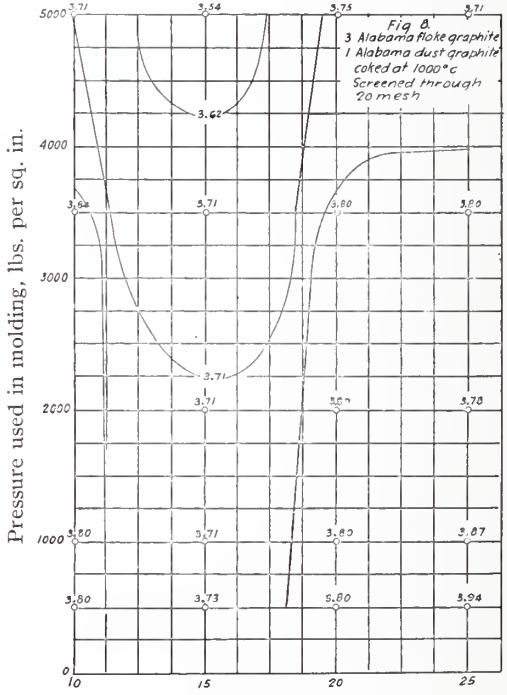
proximately 20 per cent for the lowest briquetting pressures in order to give the most desirable results.

With the mixture of flake and dust graphite, Series 2, maximum crushing strength, minimum porosity, and minimum bulkiness for each briquetting pressure occur with 15 per cent tar binder. Maximum density occurs with 10 per cent tar at 5000 pounds briquetting pressure and with 15 per cent tar at 500 pounds pressure.

High compressive strength, high density, low porosity and low bulkiness are the properties most desired. These were obtained with the highest briquetting pressure employed (5000 pounds per square inch) and for any definite addition of tar, were most nearly attained with 15 per cent tar binder.

A comparison of Series 1 and 2 at 5000 pounds briquetting

Effect of tar and pressure on the bulkiness of coked Ala. graphite.



pressure and 15 per cent tar content shows that the mixture of flake and dust gave higher compressive strength and higher porosity than the flake graphite and approximately the same density and bulkiness as the flake graphite.

Two hundred pounds of the coked graphite was prepared for making crucibles. This consisted of three parts No. 1 Alabama flake of No. 20-mesh size and one part 100-mesh graphite dust.

Fifteen per cent of coal tar by weight was selected as the binder and the briquettes were formed under a pressure of 5000 pounds per square inch. After coking at 1000° C the briquettes were crushed and screened between 16- and 100-mesh sieves.

The Alabama flake graphite has a decidedly greasy feel, while the "slippery" feeling of the Ceylon is much less. As compared to the flake and Ceylon, the coked grain appears harsh and the greasy feel is almost lacking. As compared to the Ceylon grain, the coked grain is short and "chunky," whereas the Ceylon grain is longer and narrower.

Two series of crucibles were made, one containing variable amounts of Ceylon and No. 1 Alabama flake graphite, and the other different per cents of Ceylon and coked Alabama graphite.

The crucibles containing all Alabama graphite molded with difficulty. Owing to its slipperiness, the mixture seemed to stick to the jigger tool and slide rather than pack against the mold. It did not seem to weld readily and laminated badly and when removed from the mold showed numerous welding seams. The Ceylon graphite crucibles molded much better than the flake mixture but showed a slight tendency to laminate and an occasional welding seam. The coked graphite molded perfectly, showed no tendency to laminate and no welding seams. The crucibles came from the molds apparently flawless as far as molding properties were concerned.

The crucibles were tested in a large brass foundry on two different grades of brass. Contrary to predictions, the all Alabama flake crucibles gave better service than the coked graphite crucibles, and both the coked and flake graphite crucibles gave better service than the Ceylon crucibles. The results are interesting though not conclusive—since only four crucibles of each mix were tested.[1]

Bureau of Mines,
Mining Experiment Station,
Columbus, Ohio.

[1] For more complete data regarding tests of these crucibles see "Behavior Under Brass Foundry Practice of Crucibles Containing Ceylon, Canadian and Alabama Graphites." *J. Am. Ceram. Soc.*, **2**, 208 (March 1919).

THE PROGRESS OF VITRIFICATION AND SOLUTION IN SOME PORCELAIN MIXTURES.

By Arthur S. Watts.

Introduction.

Investigators in the field of porcelains have generally held the view that the development of desirable properties is evidenced by, first, the development of sillimanite, and second, by the prog-

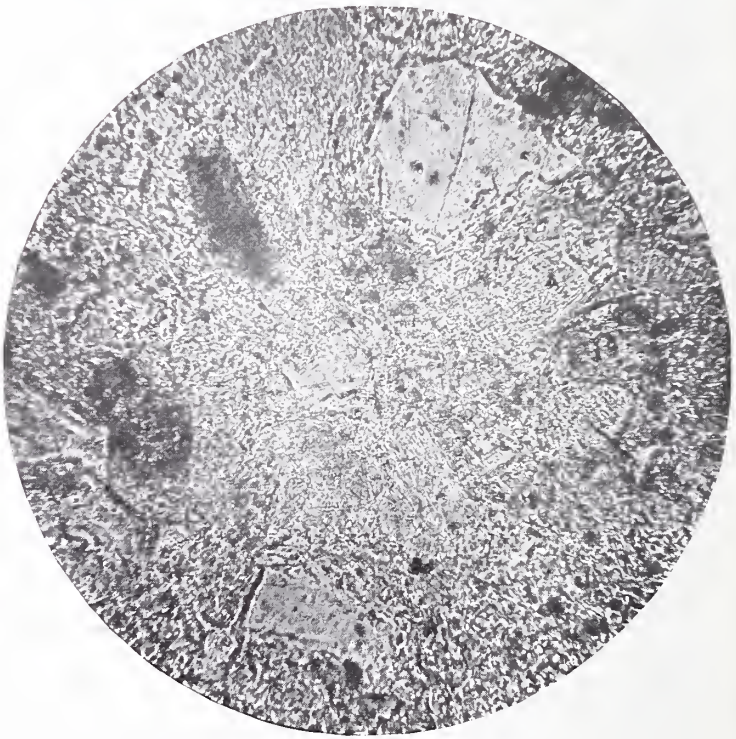


Fig. 1.—Body "W," fired to cone 9

ress of solution of the quartz grains. Both of these processes are possible of study by the use of the microscope.

In common with others, we have spent considerable time and

money in the microscopic study of porcelains but with mediocre success. Some bodies that under the microscope appear very unpromising, have proven by physical tests to be of the highest grade, while other bodies, showing great promise according to the microscopic classification, have failed completely in service. From this it would appear that we are either failing to observe

FIG. 2.—Body "W," fired to cone 11.

some vital step in the development as evidenced by the microscopic study or that certain changes are not apparent under the microscope.

Investigation.

In order to study the progress of sillimanite development and quartz solution, we prepared a body, designated "W," containing 33

per cent Canadian feldspar, 17 per cent flint, 25 per cent American china clay, and 25 per cent American ball clay. After dividing into three equal portions, one was fired to cone 9, one to cone 11, and one to cone 13. Micro-photographs of these specimens are shown in Figs. 1, 2 and 3.

A study of the specimen fired at cone 9 (Fig. 1) impresses one

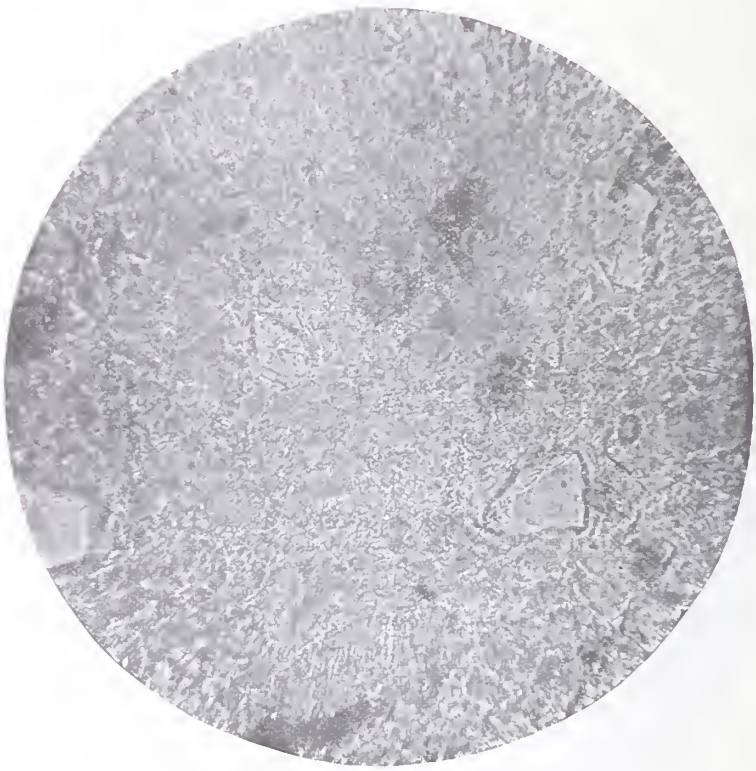


FIG. 3.—Body "W," fired to cone 13.

with the sharpness of outline of the angular quartz particles and the more or less circular particles of the larger feldspar grains, each of which is filled with fine needles of sillimanite. Other limited areas of sillimanite also appear but these are not distributed throughout the entire mass.

A study of the specimen fired at cone 11 (Fig. 2), shows the

progressive solution of the quartz particles, which have lost their corners, and the complete solution of the feldspar particles. The development of sillimanite is also much more distinct both in the number and size of crystals. It is worthy of mention that at this temperature this body attained its maximum mechanical

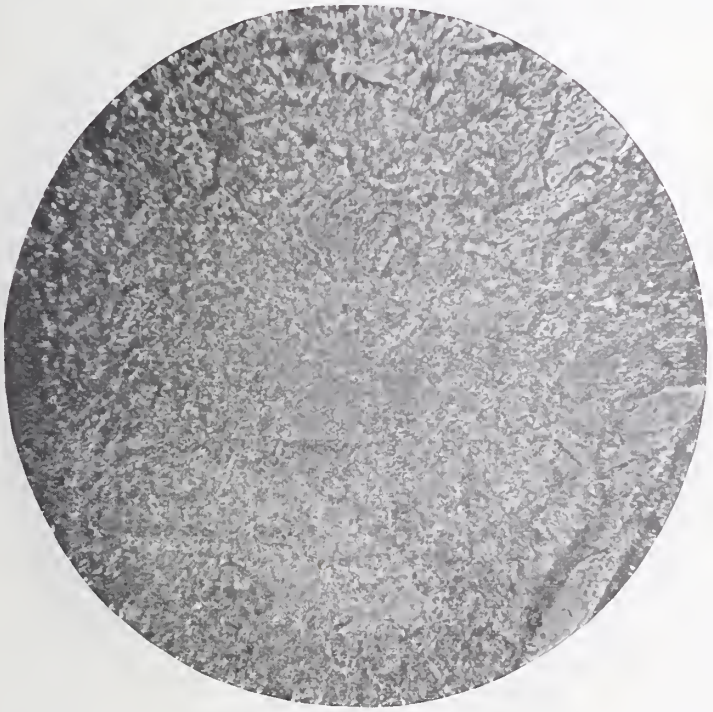


FIG. 4.—Body "O"

strength and had a high dielectric strength. It was also non-absorbent.

A study of the specimen fired at cone 13 (Fig. 3) indicates a distinct change in the appearance of the body. The quartz grains are completely dissolved on the edges. The remainder of the mass, however, has taken on a decidedly milky appearance and the sillimanite crystals, while they have increased in amount and appear throughout the entire matrix, are so clouded by the matrix that they might easily be overlooked. The dielectric

strength has slightly increased but the mechanical strength is slightly less than that of the same body when fired to cone 11.

In the study of this body, fired at the different temperatures, it may be worthy of note that the cavities (Figs. 1, 2 and 3) which appear as dark areas approach circular shape as the temperature of firing increases. It is also worthy of note that the

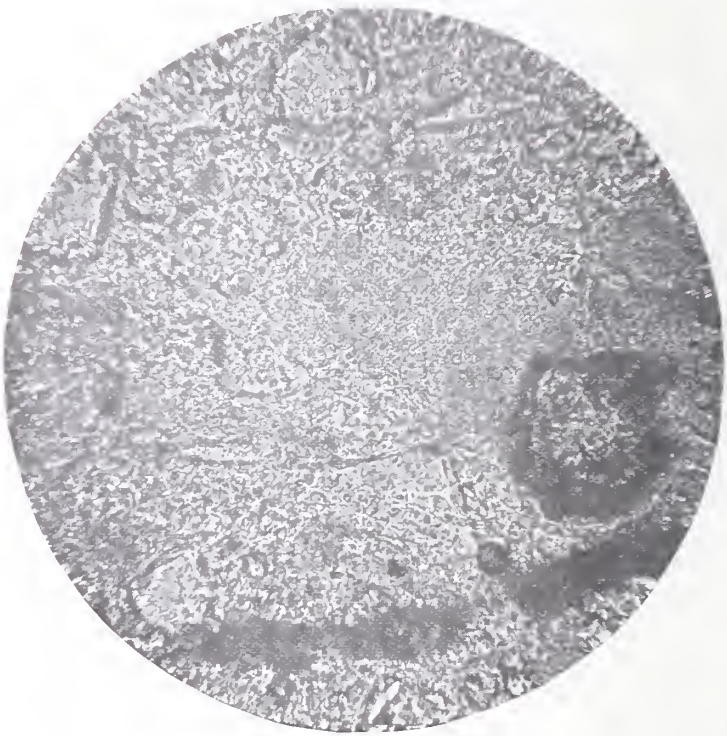


FIG. 5.—Body "L."

size of the sillimanite crystals does not increase with increase of temperature. This may be explained by the increased solubility of the clay substance in the matrix which maintains a high state of viscosity at elevated temperatures and retards crystal development. If our information regarding the solubility of quartz is true, however, the amount of quartz present should tend to overcome this viscosity by its increased solubility in the feldspar.

This raises the question of the influence of the degree of viscosity of the solvent upon the sillimanite development. In

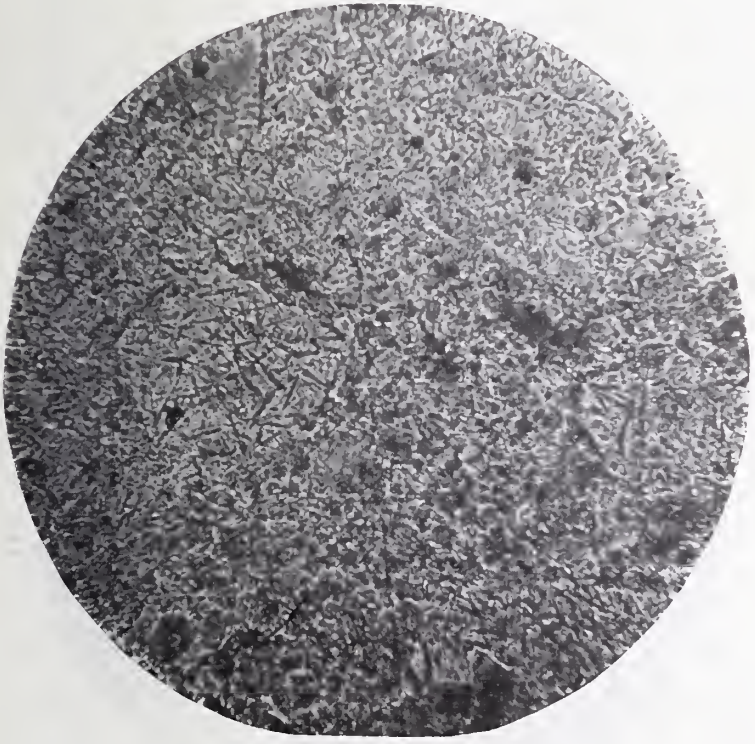


FIG. 6.—Body "V."

studying this phase of the subject a series of bodies having the following compositions were prepared:

	"O" Body. Per cent.	"L" Body. Per cent.	"V" Body. Per cent.
Potash feldspar	21.35	21.35	...
Soda feldspar	...	...	21.35
Whiting	...	1.92	1.92
English china clay	42.93	42.93	42.93
English ball clay	8.00	8.00	8.00
Flint	26.73	26 73	26.73

These bodies were fired to cone 12 under oxidizing conditions.

Body "O" (Fig. 4), which contains no flux except potash feldspar, shows sillimanite development to about the same degree as was observed in the all-American body when fired at cone 9. The quartz grains, while smaller in size, also show about the same degree of solution as in the cone 9 body.

Body "L" (Fig. 5), which contains about 2 per cent whiting

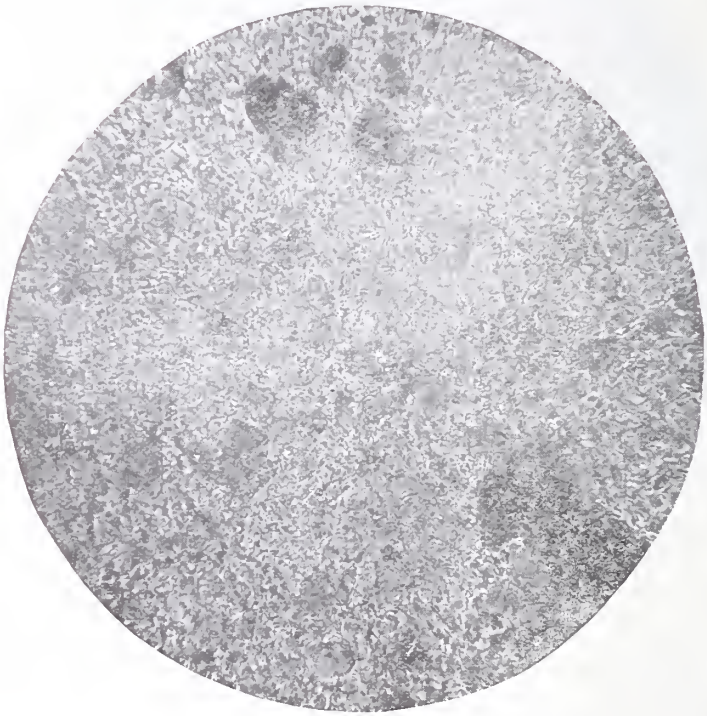


FIG. 7.—Body "Q."

as flux in addition to the potash feldspar, does not display any increased sillimanite development, but the matrix is noticeably more glossy and the quartz grains show slightly more solution.

Body "V" is exactly the same as body "L," except that soda feldspar was substituted for potash feldspar. This substitution had a marked effect both in the degree of quartz solution and sillimanite development (Fig. 6). The quartz grains have gone into solution to a degree far in excess of that attained in any

of the other bodies studied. It is noticeable also that the sillimanite crystals are at least five times as large as those in any other body studied. The distribution of sillimanite, however, is not more general than in the potash-feldspar bodies. The matrix is perhaps somewhat more glossy and numerous small spherical cavities are noted—indicating the development of a vesicular structure.

Another interesting and instructive study was made and is here presented by Body "Q" (Fig. 7). This body is of the same composition as body "L" (Fig. 5), but differs in that the feldspar, whiting, and a portion of the clay and flint were previously fused and reground thus rendering them presumably more active as fluxes. This body was fired to cone 10. The degree of solution is distinctly apparent in the quartz grains remaining and the crystals of sillimanite are more sharply defined and of slightly increased size over those in Body "L," although much smaller than those present in Body "V." The most striking improvement is in the transparency of the matrix. The presence of large numbers of tiny spherical cavities indicate that this body was over-fired even more than was the case with Body "V."

Summary.

The foregoing data indicate that the development of sillimanite starts in the feldspar grains and progresses as fusion progresses. The matrix of fused feldspar saturated with clay substance and quartz permeates the mass—the whole mass thus becoming more homogeneous.

The introduction of the calcium flux does not apparently add to the sillimanite development, although it undoubtedly increases the solution of the quartz grains at any given temperature.

The substitution of soda-feldspar for potash-feldspar is accompanied by increase in size of the sillimanite crystals but the amount of sillimanite does not apparently increase.

The preliminary fusion of the feldspar and calcium flux before incorporation in the body adds to the fluxing action and tends to increase the size of the sillimanite crystals developed. It also aids the solution of the quartz grains but the amount of sillimanite developed is not materially increased.

Conclusions.

The solution of quartz in feldspar or feldspar-calcium glass is apparently proportional to the degree of fusion and the viscosity of the mass.

The sillimanite development in a porcelain is evidently the result of a high state of saturation of an alkaline-aluminum-silicate. While the potash-feldspar porcelain shows the largest amount of sillimanite, this may be explained by the fact that the soda-feldspar used contained a larger amount of magnesia than did the potash-feldspar used. The addition of calcium undoubtedly develops a calcium-aluminum-silicate which does not encourage the development of sillimanite. It is reasonable to assume that this calcium-aluminum-silicate has a distinct solvent action on the clay substance and the natural result would be sillimanite development—if the same laws hold for all fusions saturated with clay substance. The fact that all of the clay substance is not dissolved would indicate that the fusion containing calcium has either not reached a state of saturation or that the presence of the calcium-aluminum-silicate is unfavorable to sillimanite development.

A study of natural rocks in which sillimanite and its allotropes occur would indicate that the presence of the alkaline earths is unfavorable to sillimanite development. For example, cyanite, andalusite and sillimanite, all of the composition $Al_2O_3.SiO_2$, are found in nature only in rocks of high silica content and associated with secondary minerals containing an alkali, alumina and silica. The chief of these are muscovite ($K_2O.Al_2O_3.6SiO_2.2H_2O$), paragonite ($Na_2O.2Al_2O_3.6SiO_2.H_2O$) and lepidolite ($KLiO.Al_2O_3.3SiO_2.H_2O.F$). Thus we see that the rocks containing sillimanite and its allotropes are high in alumina and alkali. Selective crystallization has enabled the alkali present to claim its maximum amount of alumina and the alumina excess enters into combination with an equal molecular amount of silica to form sillimanite or one of its allotropes.

There is no reason why this same process should not operate in the development of porcelain. The clay substance when once broken up by the fused feldspar would first saturate the feldspar and

the excess alumina would combine with an equal molecular amount of silica to form sillimanite. As in nature, we do not find sillimanite or its related minerals developing in rocks containing alkaline earth, so in the porcelain we find the presence of calcium acting in a manner unfavorable to sillimanite development.

The industrial application of porcelain requires that three questions be answered, *viz.:*

(1) How may sillimanite be developed in a porcelain so that the highest grade of product will result?

(2) To what extent can sillimanite be developed in a porcelain without the loss of any desirable physical property?

(3) To what extent can the solution of quartz progress to the benefit of the porcelain?

Some light has been thrown on the first question by this study, but much remains to be done.

The loss in mechanical strength displayed by Body "W," when fired at cone 13, indicates that while sillimanite is an important constituent, its development is not always an evidence of improved physical properties.

A simple study reported by the writer indicates that incorporation of the flint into a calcine or semi-frit before introduction into the porcelain increases its resistance to shock, but such limited research is not a safe basis for a general statement and an exhaustive study is required before any general conclusions can be safely drawn.

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AN APPARATUS FOR STUDYING THE DISSOCIATION OF CARBONATE ROCKS.

By. G. A. Bole.

Introduction.

It is common knowledge that a static determination of dissociation pressures not only requires complicated apparatus, but also a great amount of experience and painstaking manipulation to obtain even fairly accurate results.

The results of carefully carried out determinations of the temperature at which the pressure of CO_2 becomes atmospheric vary between 812° C and 915° C, for calcium carbonate, depending on whether the work of Le Chatelier or Zavriev be considered. The results of the work of other careful investigators, such as Johnston, Potts, and De Bray, fall somewhere between these two extremes, each claiming special accuracy for his work.

The disadvantage in depending on a lag in a time-temperature curve for dissociation temperatures is apparent, when one considers the fact that different parts of the sample vary in temperature from 50 to 100° C, and that the CO_2 liberated is in equilibrium with only that CaO which is at the lowest temperature. The more slowly the temperature is raised, the more accurate the results, however, and it can be said in its favor that the method approaches the conditions under which the ceramist works.

Description of the Apparatus.

For use in our laboratory we have devised a very simple piece of apparatus which has given satisfactory results even in the hands of student manipulators. It has been used not only for determining the temperature at which the pressure of the products of dissociation becomes atmospheric and for deriving the data for plotting dissociation curves of carbonate rocks, but also for determining the rate of dissociation of carbonates at different temperatures. It is of value for control work in calcination processes and for the analysis of magnesian limestones, where

it is required to distinguish between the CO_2 combined with the magnesia and that in union with the lime.

The apparatus is in effect a Victor Meyer vapor-density apparatus, in which the steam jacket has been replaced by an

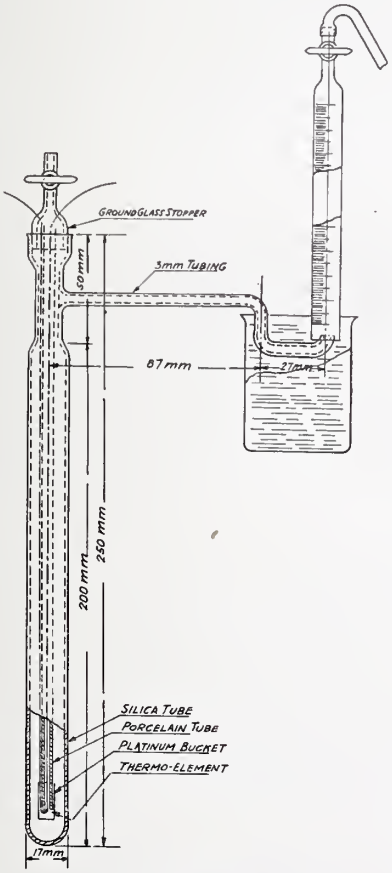


Fig. 1.

electrical tube furnace. The ends of this are well insulated to insure an even heat. The inner tube is of clear fused silica. This latter, shown in Fig. 1, is 10 inches long and $^1/_2$ inch in diameter at the larger end. Eight inches from the closed end it is somewhat

constricted in order that an asbestos disc may be introduced, to keep the hot gases from coming in contact with the rubber stopper —in case such a stopper is used instead of one of ground glass. Half way up the constricted portion a delivery tube is fused in. This is bent in such a fashion as to lead into a water- or mercury-trough. The open end of the inner tube is closed by means of a ground glass stopper, into which are fused the wires of a thermo-element, and also a tube containing a ground-glass stopcock. The gases evolved through the side delivery tube are measured over water, saturated with the products of the dissociation, or over mercury in an inverted burette which has been accurately calibrated.

As few joints as possible are desirable in order to insure against leakage. The parts of the tube outside the furnace are small, thus obviating so far as possible any expansion or contraction of the gas within the tube. Determinations should be carried out in a room of even temperature and devoid of drafts. The expansion and contraction of the silica tube itself is negligible. The whole system is tried out against 40 to 50 mm. of mercury before each determination, to guard against a faulty insertion of the stopper.

It is of importance that the tube be inserted into the furnace to the same depth each time. This is insured by making a mark on the tube, or by having a fire-clay bench built into the furnace beyond which the tube cannot enter.

The sample (0.1 gram) is contained in a small platinum bucket which just fits over the end of the porcelain insulation tube of the thermoelement, where it is held in place by means of a splinter of porcelain. This also serves to insulate the thermoelement leads from the bucket.

The temperatures are read from either a standardized millivoltmeter or from a potentiometer-galvanometer arrangement. A thermoelement is placed in the furnace also, in order that the temperature control may be more accurate. The thermoelements are standardized against authentic transition-temperatures of pure chemical substances.

Making the Determinations.

Dissociation Curve.—When securing the data for plotting a dissociation curve of a magnesian limestone, for example, the process is as follows: The sample is introduced into the quartz glass tube and is then put into the furnace. The temperature is run up to 450° C with the stopcock open, in order to drive out any moisture. It is then closed and the temperature raised through equal temperature intervals in equal time. The volume of the gas evolved is measured at the end of each time interval. The determination is continued without interruption until the dissociation has gone to completion, that point being indicated by the volume of gas evolved. The thermometer, which is kept in the water, is read after each time-interval to ascertain the temperature of the gas which has bubbled through it. The gas evolved is run through absorption pipettes, and if any carbon monoxide is found to be present, the sample is treated for removal of organic matter and a new determination is made.

The gas is evolved at atmospheric pressure, but as it ascends the tube, displacing the water, the bubbles increase in size—due to reduced pressure. Therefore, after each reading, the height of the water must be measured and subtracted from the barometric pressure, together with the vapor pressure of the water for the temperature at which the determination is being carried out. Part of the gas evolved is due to expansion of the air in the tube, and part to the products of dissociation, so it is necessary to run a blank with the platinum bucket empty, which gives the expansion curve. This curve when plotted on the same sheet as the total volume curve enables one to see the true dissociation, or the air expansion for each equal temperature interval may be subtracted from the total volume of gas evolved during that interval, and plotted thus against time.

Dissociation Temperature.—When determining the temperature at which the pressure of the products of dissociation just overcome the atmospheric (dissociation pressure) it is necessary only to find at what point the gas begins to be evolved when the temperature is held constant. The procedure is as follows: The sample and apparatus are arranged as when securing data

for a dissociation curve. The temperature is increased with the stopcock closed until a rapid evolution of gas indicates that dissociation has begun. The temperature is allowed to fall until bubbles cease to be evolved. The stopcock is then opened to equalize the pressure within the tube. If no gas comes off when the cock is again closed, the temperature has fallen below the dissociation point.

The furnace is again heated till evolution of gas sets in. It is then allowed to fall very slowly, and the stopcock opened from time to time until a point is reached where gas just ceases to come off. This temperature is held for thirty minutes and if no gas is evolved it is evident that this point lies just below the dissociation temperature. The same operation is repeated, holding the temperature five degrees higher than previously. If gas is evolved at this point it is evidently one slightly above the dissociation temperature. Carrying out this method carefully one can determine the temperature of dissociation as accurately as the temperature reading device permits, generally from 2 to 5° C.

Rate of Dissociation.—When the rate of dissociation is being determined the procedure is somewhat modified (See Fig. 2). The sample (0.5 gram) is weighed in the platinum bucket which is placed in the neck of the quartz-glass tube, being held in place by a wire which slips through the glass stopcock. Since it is impracticable to have a thermoelement inside the tube while running these determinations, the quartz tube is placed in a larger porcelain tube, in the bottom of which the thermoelement is inserted. The space between the two tubes serves as a constant temperature bath.

The arrangement is then placed in the furnace and the temperature brought up to about 50° C above the point at which the determination is to be made. It is allowed to fall slowly until the desired temperature is reached, where it is held for fifteen minutes. In this way it has been found that the temperature within and without the tube never varies more than 5° C. The bucket is now released and allowed to fall to the bottom of the tube. The stopcock is closed so that the bubbles evolved, due to dissociation, ascend in the measuring burette. There is a sudden evolu-

tion of gas, due to some of the hot gases being driven to the upper part of the tube and the colder gases descending, but there is an equal subsequent contraction as the temperature of the tube again adjusts itself to that of the furnace. The time at which the stop-cock was closed is noted, and the reaction is allowed to proceed for a

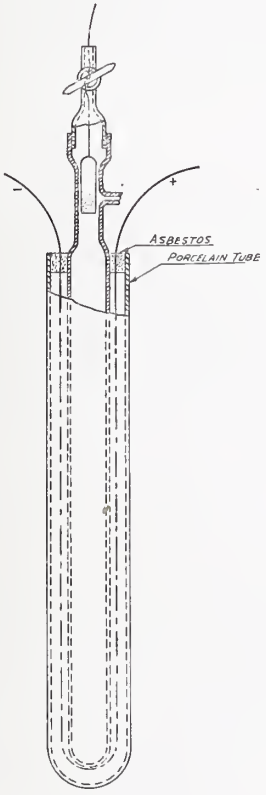


Fig. 2.

definite period, after which it is opened to the atmosphere and the volume of evoluted gas read. The bucket is withdrawn, a new sample of exactly the same weight introduced, and a determination run in the same way at a higher temperature. When sufficient points have been determined the corrected volumes are plotted against temperature.

Quantitative Analyses.—The apparatus has also been used to determine the carbon dioxide combined with the magnesia and that combined with the lime in a partially calcined dolomite. The procedure requires that the temperature be run up to 750° C with the sample suspended in the neck of the tube. When the temperature has become constant the bucket is released and the gas evolved at that temperature is measured. The temperature is raised to 900° C, where the CO_2, due to dissociation of $CaCO_3$, will be given off rapidly. The previously determined expansion, due to the rise through 150°, is subtracted from the reading at 900° C.

Carbonate rocks can be analyzed quantitatively for magnesian and lime CO_2 in the same way. MgO and CaO calculated from the CO_2 evolved at the two temperatures give results that do not vary from the average of several gravimetric determinations in a greater degree than the gravimetric determinations among themselves when fairly pure dolomites are used.

Advantages and Conclusions.

(1) A determination can be run in a short time.

(2) Impurities such as organic matter, occluded gases, and moisture can be driven off just before a determination is run, by simply manipulating a stopcock.

(3) The pressure can be equalized repeatedly during a determination by opening a stopcock. In this way metastable equilibrium and excess pressure on the apparatus are avoided.

(4) Working at the same pressure at all times (atmospheric) the inaccuracies of static measuring instruments, and substances used therewith, are avoided.

(5) The filling of the apparatus is simple, and the accuracy of the temperature is assured.

(6) The use of a transparent tube enables one to ascertain with certainty whether the thermoelement is actually in its proper position, both before and after a determination.

(7) The evolved gas can be analyzed. Thus one can be sure of the dissociation products being purely those of the reaction: $RCO_3 \longrightarrow RO + CO_2$.

The apparatus may be used for several processes:

(1) Determination of the temperature of dissociation.

(2) Determination of the rate of dissociation.

(3) Control work in calcination processes.

(4) Analyses of magnesian limestones, when it is required to distinguish between the CO_2 combined with the CaO and that combined with the MgO.

N. Y. State School of Ceramics,
Alfred, N. Y.

AMERICAN CERAMIC SOCIETY.

Acquisition of New Members During May, 1919.

Associate.

Ashman, Alfred O., 277 Delaware Avenue, Palmerton, Pa.
Boudouard, M., 292 Rue Saint Martin, Paris, France.
Corrigan, F. S., Sheet Metal Products Co., Toronto, Canada.
Ellinwood, R. S., Hemingray Glass Co., Muncie, Ind.
Haberstroh, H. N., 53 W. Jackson Blvd., Chicago, Illinois.
Harrell, E. R., Supt., Graham Glass Co., Checotah, Okla.
Hersh, Lewis E., 370 Delaware Ave., Palmerton, Pa.
Holland, Job., Box 22, Sheffield, England.
Mumma, C. M., Lock Box E, Haviland, Ohio
Niver, R. W., Thatcher Mfg. Co., Elmira, N. Y.
Patch, James B., Whitall-Tatum Co., Millville, N. J.
Strommer, L. T., Beaver, Pa.
Tiebout, Cornelius H., Jr., Gleason-Tiebout Glass Co., 99 Commercial St., Brooklyn, N. Y.
Whittemore, O. J., Iowa State College, Ames, Iowa.
Yingling, Walter A., Supt. Woodbury Glass Co., Winchester, Ind.

Corporation.

Illinois Glass Co., Alton, Ill.

ACTIVITIES OF THE SOCIETY.

May 9th. In accordance with the recommendation of the Division of Chemistry and Chemical Technology of the National Research Council, it was voted to approve the admission of neutral countries to the International Chemical Council.

May 9th. The University Bank, Alfred, New York, was approved as the depository for the funds of the Society by the Secretary each month.

JOURNAL
OF THE
AMERICAN CERAMIC SOCIETY

A monthly journal devoted to the arts and sciences related to the silicate industries.

Vol. 2 **June, 1919** **No. 6**

EDITORIALS.

THE FIFTH CHEMICAL EXPOSITION.

Those who were in attendance at the Fourth Chemical Exposition in New York last year will require little urging to take them to the Fifth Exposition to be held in the Coliseum and 1st Regiment Armory at Chicago, the week of Sept. 22nd. The Chemical Exposition has come to be one of the most important industrial events of the year. At the Exposition of last year were shown achievements of American industry in meeting war demands and in the development of sources of supply to replace foreign materials unavailable under existing conditions. At the Exposition of this year will be shown many of the advancements made since a year ago, in addition to some which have hitherto been undisclosed to the public.

The ceramic industry, although one of the oldest manufacturing industries, is at the same time one of the least known of the great technical industries. The representation of the American Ceramic Society at the past two Expositions has gone far towards bringing the importance of the industry to the attention of the public and related industries. Plans are well under way for a suitable representation of the Society at the Chicago Exposition and it is hoped that a goodly number of our members, particularly those in the West, will take advantage of this opportunity to get together and study the advances made in their own and related industries.

STANDARDS FOR MATERIALS.

We have repeatedly heard statements to the effect that American clays and other pottery materials are subject to undue varia-

tions in quality from one shipment to another or even in the same shipment. This is said to be particularly true of ball clays and kaolins. Although some improvement in quality has been noted it would appear that the exercise of greater care in the selection and preparation of clays for the market would lead to a better understanding between the clay producer and user. However, the clay users are partly responsible in many cases for the poor quality of clays which are delivered to their plants. Very often, little or no attention is paid to the condition and quality of the clay as it is delivered, and it is only when losses occur in manufacture that complaints are made as to the quality of the clays. The simple and obvious tests which should be applied to every shipment of clay received are too often neglected, or the tests applied are inadequate to a determination of the acceptability of the clay for the purposes used. Tests as to moisture content, shrinkage, fineness, color and cleanliness should be made upon each shipment of material received. We know of plants where no attention is paid to the moisture content of the clays and other materials which go into the body mixtures. A variation of 10 per cent in the moisture content of a clay is not unusual and unless this is taken into consideration in the preparation of the batch the proportions of the mixture will be thrown out and a uniform product will not result.

Once the consumer has established standards for his materials the producer will very quickly endeavor to meet those standards. As it is now, the producer is not to be greatly blamed for the quality of his product. In many cases most anything will be accepted without question—in fact, he does not know just what is required of his materials and just what are the allowable variations in any one particular. The adherence to rigid specifications for the raw materials is obviously the first step in the standardization and improvement in quality of our ceramic wares.

CERAMIC ABSTRACTS.

Starting with this number, abstracts of ceramic literature will be published monthly in the Journal. Ceramic literature is so widely diversified and scattered that considerable difficulty

has been experienced in the classification and assigning of the work so as to thoroughly cover the field. Once a full corps of abstractors has been secured it is planned to cover the entire field of literature on all ceramic subjects, including clays and related products, glass, cement, lime and ceramic patents. The articles will be abstracted quite fully and in such a way as to render them easily readable by most of our members.

THE SUMMER MEETING.

Attention is called to the announcement of the Summer Meeting on page 503 of this number. Owing to a change in plans the meeting will convene on Monday morning, August 4th at the Hotel Iroquois, Buffalo, instead of Monday evening at the Hotel Statler, as announced in the May number of the Journal. An inspection trip for Monday afternoon is being arranged. Monday evening will be reserved for Professional Division meetings and a general business meeting of the Society. Tuesday is reserved for a trip to Niagara Falls and on Wednesday, at least three industrial plants will be visited in Buffalo. Wednesday evening the party will take the nine o'clock boat for Cleveland where the remaining sessions will be held.

ORIGINAL PAPERS AND DISCUSSIONS

PROCEDURES IN THE MANUFACTURE OF OPTICAL GLASS.

By W. S. Williams and C. C. Rand.[1]

Introduction.—The Bureau of Standards began experimental work on the production of optical glass in July 1914, and investigations along this line are still in progress. The present paper is intended as a brief statement of the general problem of making optical glass. A more thorough discussion of the processes involved and of the results obtained will be given in a later paper or bulletin.

Requirements for Optical Glass.—Optical glass is that type which is of a quality suitable for use in precision instruments such as range finders, gun sights, telescopes, binoculars, periscopes and camera lenses. The requirements for these purposes are very exacting. The most serious defect is inhomogeneity. This may be manifested as differences in the refractive index in different parts of the melt, or by streaks or thin sheets of different index from the surrounding glass. The latter condition is by far the most troublesome.

By the use of suitable apparatus, irregular deviations of light rays which are caused by defects variously known as "striae," "veins," "cords," "waves," etc., may be detected. These may occur in threads or sheets, or in wide diffuse bands either straight or curved and sometimes looped and twisted. The most powerful and delicate precision instruments are made with glass entirely free of striae and practically free of stones. For certain instruments, however, a few small striae or seed are permitted

[1] By permission of the Director, Bureau of Standards.

as their presence will cause no appreciable harm. In the final annealing all strain must be eliminated. Devitrification, cloudiness or crystallization will cause the rejection of the glass. Hygroscopic or tarnishable glass cannot be used in instruments which are to be exposed to weathering conditions. Furthermore, the product must have sufficient strength to prevent breaking in the grinding, polishing and mounting operations.

The raw materials and the melting pots must be free from such impurities and elements as will color the glass and thus increase the absorption of light. However, other characteristics are at times so desirable that light absorption becomes a secondary consideration, as in special flints and dense barium crowns. Compositions and melting operations should be coördinated and standardized to produce, in successive melts of the same composition, glasses which are as constant as possible in optical properties, such as the refractive index, dispersion and density. It is largely upon these values that the optician makes his choice and calculates his lens elements accordingly. It even becomes necessary in some optics to recompute formulas for slight changes in these values between different melts, and even to reconstruct grinding and polishing tools.

Production of Optical Glass.—The exacting requirements, the small market for the product, and the prejudice in favor of imported glass, have been the causes of the small production of optical glass in the United States. A great deal of research was necessary before production could be assured. Manufacturers were reluctant to spend money on research without any assured profit, and in this case there was no promise of large returns. In spite of these unfavorable factors, several glass manufacturers are reported to have attempted quantity production of optical glass in America. About 30 years ago an attempt was made at Lenox, Mass., but it was abandoned. About the time of the Spanish-American war some very good optical glass was made in the Macbeth factories at Elwood, Ind. In 1912, the Bausch & Lomb Optical Company built an experimental plant at Rochester, N. Y., and with the aid of an experienced man began production.

With the beginning of the war in 1914 came a great demand for instruments, glass stocks were quickly depleted and America realized the folly of depending on Europe for its optical glass. Since practically all naval and military operations depend on accurate optical instruments, it was imperative to take such measures as would secure an ample supply to meet the great and rapidly increasing demand. In 1914 the Bureau of Standards began experimental work on the production of optical glass. When the United States entered the war a number of manufacturers began the production of optical glass, some of them on quite a large scale. Among these were: The Pittsburgh Plate Glass Co., The Keuffel and Esser Co., The Hazel-Atlas Glass Co., The Spencer Lens Co., The Carr-Lowry Glass Co., and the H. C. Fry Glass Co.

Many errors and failures were naturally to be expected during the experimental stages of development. The ground work was based on the chemical analyses of European glass and a meager knowledge of European technique.

Raw Materials.—One of the first problems to be solved was that of securing pure raw materials. The very nature of the product demands a degree of purity unknown in other lines of glass manufacture. Propagandists had been very active in attempting to convince America that optical glass was a product altogether too difficult to be attempted here. Well known glass sands were found to contain enough iron to condemn them for optical glass, where freedom from color is a requirement, and no decolorizers such as manganese and selenium can be used. Sands were found, however, which were sufficiently pure and furthermore had the requisite size and uniformity of grain. The sands used were readily available and obtainable at a fair price. With the exception of the removal of magnetic iron, they were used just as received from the shippers without refinement or further treatment. This also holds true for all other materials used in manufacturing optical glass at the Bureau of Standards. Typical screen tests of sand used are reported in Table 1. The maximum and minimum percentages of impurities found in the materials used are given in Table 2. The color and transmissions

obtained with these materials, melted in a porcelain pot, compare very favorably with those published in the price lists of the foreign makers.

TABLE 1.—TYPICAL SCREEN TESTS OF SAND.

Screen size.	Per cent remaining on screen.
35	2.47
65	82.59
100	8.76
150	3.90
200	1.77
Through 200	0.43

TABLE 2.—IMPURITIES FOUND IN MATERIALS

	Fe_2O_3.		SO_3.		Cl.	
	Max.	Min.	Max.	Min.	Max.	Min.
Barium carbonate.....	0.009	0.0005	0.12	0.0774	0.13	0.01
Barium carbonate, C. P.....	0.0067	0.004	0.08	0.018	0.069	0.0124
Red lead.....	0.0056	0.0015	0.055	0.01	0.13	0.017
Soda ash.....	0.003	0.0014	0.03	0.0049	....	....
Hydrated lime.....	0.048	0.026	0.167	0.06	....	....
Potash (K_2CO_3).....	0.01	0.004	0.07	0.018	0.31	0.11
Zinc oxide.....	0.006	0.0057	0.327	0.11	0.35	0.078
Saltpeter (KNO_3)....	0.0019	0.0007	0.0016	....	0.07	0.05
Borax.....	0.002	0.0004	0.034	0.018	0.005	0.0034
Alumina (Al_2O_3).....	0.0022	0.0022	0.15	0.15	....	....
Sand.....	0.02	0.0148	....	....	....	....

The intimate mixing of the ingredients of the batch is very important. One of the theories advanced for the formation of striae is that the striae are of different chemical composition from the surrounding glass. Viewed in this light, it is obvious that the tendency toward striae is lessened by intimately mixing the batch. At the Bureau of Standards the batch materials are passed through a 12-mesh screen and then mixed for one hour in a ball mill using no pebbles.

Pots for Optical Glass Melting.—When large-scale production of optical glass was commenced a most serious obstacle was encountered by the failure of pots. Pots from many commercial sources were tried but the action of the glass upon them was so corrosive that it ate its way through the walls in a short time. The successful solving of this problem at the Bureau of Standards has been fully described in this Journal.[1] A covered pot was adopted because it prevents contamination from particles of the furnace crown. The glass is melted in single-pot furnaces fired by natural gas and compressed air. The greater part of the waste gases are removed through flues in the floor leading to a stack flue. In Fig. 1 are shown a part of the working drawings of this furnace.

Before drying, the inside walls of the pot should be carefully finished and the grog particles worked well back from the surface and covered with clay. This clay coating offers a resistant shield between the glass and the pot body. When the pot is too dry before finishing, the grog will not readily work back and the pot-maker is then compelled to wet the surface excessively. Fine, hair-line, surface cracks result and these may escape detection before the pot is used. The glass gains access to the pot body through these cracks causing pieces of the pot to become loosened and form "stones."

Many pot failures have been due to an attempt to use the pots before they were thoroughly dried. Still others are due to an unequal distribution of heat causing the pots to warp and crack. The drying schedule used is shown in Fig. 2 and the burning schedule in Fig. 3. It has been found that a pot may be burned so dense as to become too brittle for use. When these pots are chilled they fall apart and the melt is lost.

The practice at the Bureau of Standards' factory is to set the pot in a melting furnace on a bed of sand about three inches deep. A brick is placed under the front of the pot to permit circulation of heat thus insuring a well-burned bottom. When the furnace has reached 1100° C (Fig. 3) the brick is removed and the pot lowered into the sand bed. After the pot is vitrified

[1] A. V. Bleininger, *J. Am. Ceram. Soc.*, 1, 1–23 (1918).

the temperature is reduced, if necessary, to the melting schedule as shown in Fig. 4.

Charging the Batch.—When the melting schedule is reached 200 pounds of cullet are put into the pot. This forms a coating of glass over the bottom of the pot and protects it from the active corrosion of the raw batch. The cullet is carefully selected and

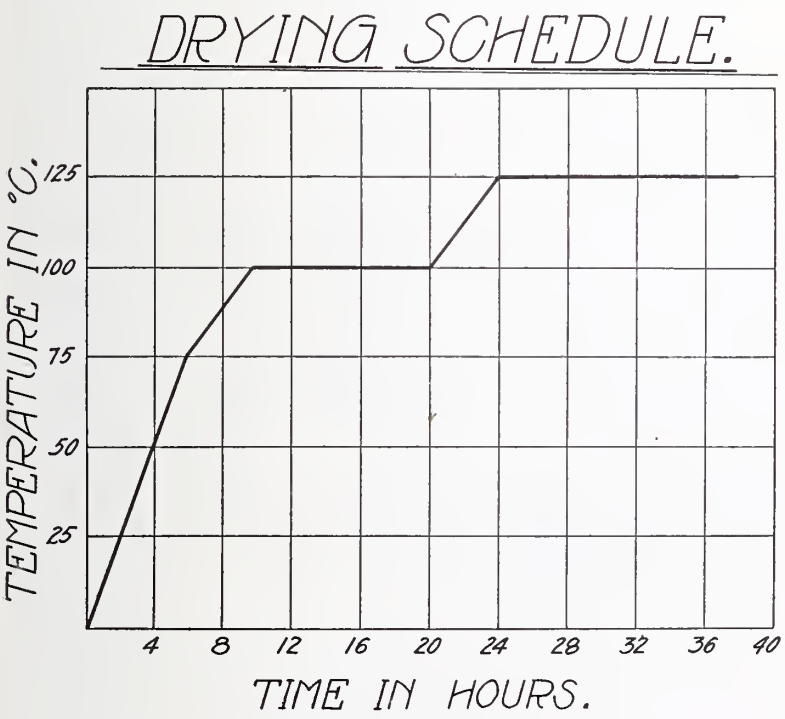


FIG. 2.

inspected for color, stones, pot-shell, sand and dirt. It is broken to nut-size and washed. When cullet is used with the raw batch it is mixed uniformly with it.

One hour after the cullet is introduced a fill of 150 to 200 lbs. of batch is made. The same amount is filled every $1^1/_2$ hours thereafter until the pot is full. During filling and fining it is important to avoid a big drop in temperature; for if the glass is chilled,

it is very difficult to eliminate the fine seeds thereafter. "Seedy" glass at this stage of the melt may also result from a lack of readily volatile ingredients in the batch. The elimination of seeds in certain types of glass was accomplished by substituting nitrates for carbonates in the batch. Seeds may also be eliminated by

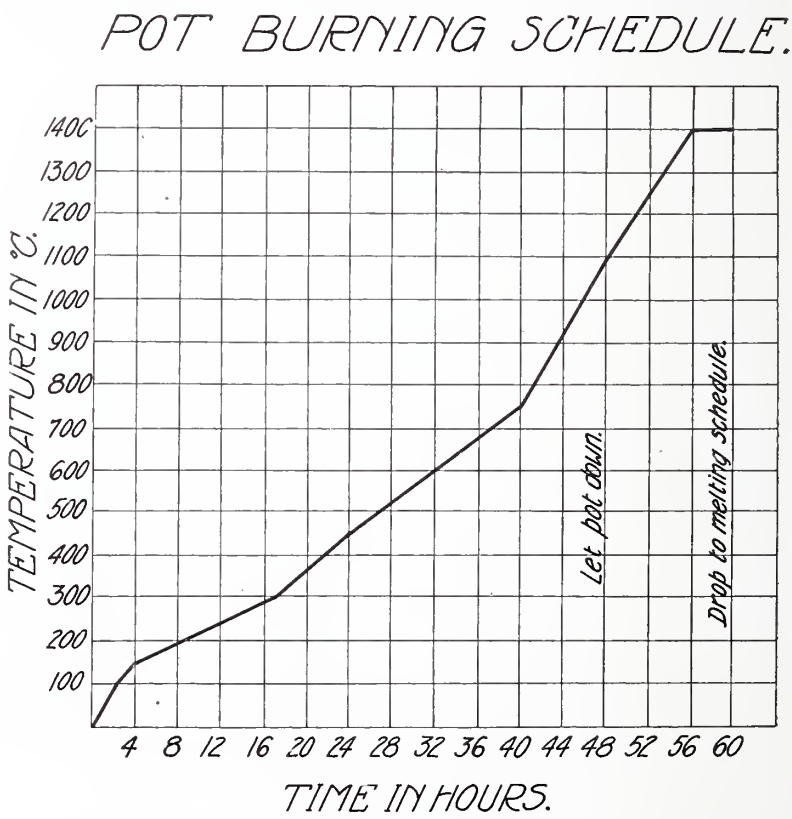


FIG. 3.

"blocking" with ammonium nitrate, which causes the metal to boil very vigorously. "Blocking" is done by wrapping about 1/4 of a pound of ammonium nitrate in paper, placing the package in a forked rod and then plunging it into the center of the metal. Violent boiling ensues, and the operator must be on guard not to become burned.

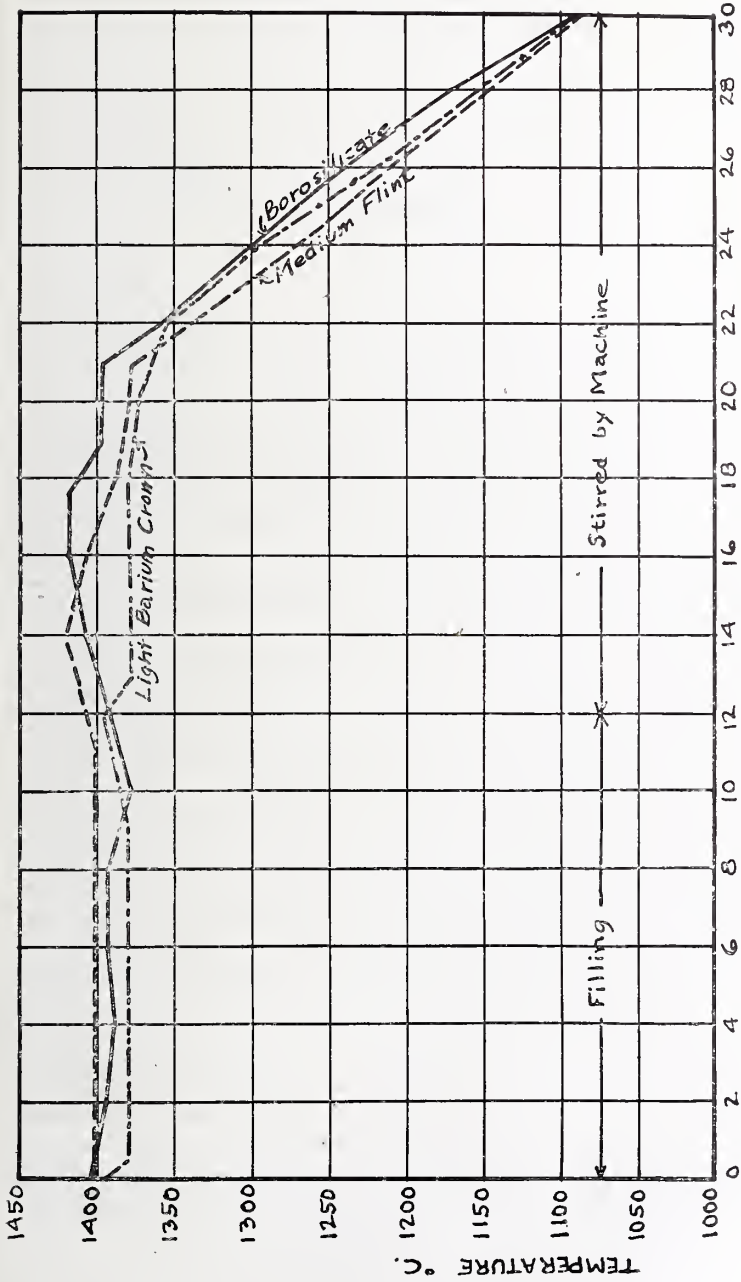


FIG. 4.—Typical time—temperature curves.

Stirring.—Two hours after the last fill a porcelain-clay stirring rod thimble is laid on the surface of the glass. This thimble, shown in Fig. 5, is cast from the porcelain pot mixture used in

FIG. 5—Stirring-rod thimble.

casting melting pots. It is heated to 1200° C and then allowed to attain the same temperature as the metal by laying on the surface of the glass for one hour prior to turning the closed end down into the metal. When in the latter position a water cooled iron rod is inserted in the open end of the thimble and stirring begins.

Typical melting and stirring schedules are shown in Fig. 4. As previously stated, the greatest percentage of rejected glass is due to inhomogeneity, or striae. This defect is overcome by stirring the metal until a homogeneous mixture is obtained. The refractive indices demanded in optical glass require compositions of materials whose densities vary widely. The heavier constituents have a tendency to segregate and settle to the bottom of the pot during filling and melting. These constituents must be stirred with the lighter ones until thoroughly incorporated with them. Furthermore, the glass adjacent to the pot walls is of a different composition from that of the center, which is due to the solvent action of the glass upon the clay walls of the pot. If this contaminated glass is stirred into the center of the melt and is not thoroughly assimilated, it will cause striae. While rotating, the stirring thimble is kept about three inches from the sides and bottom of the pot to prevent stirring the pot-contaminated glass into the center. As stirring progresses the radius of the stirring circle is continually shortened and the speed reduced. The stirring machine is shown in Fig. 6. Mechanical stirring is preferred because it insures the agitation of the entire melt, speed and radius are easily controlled, and the stirring thimble is kept away the required distance from the walls of the pot. When stirring begins, the machine is run at from 20 to 23 r. p. m. This speed is gradually reduced as the glass cools and stiffens, to prevent stir-

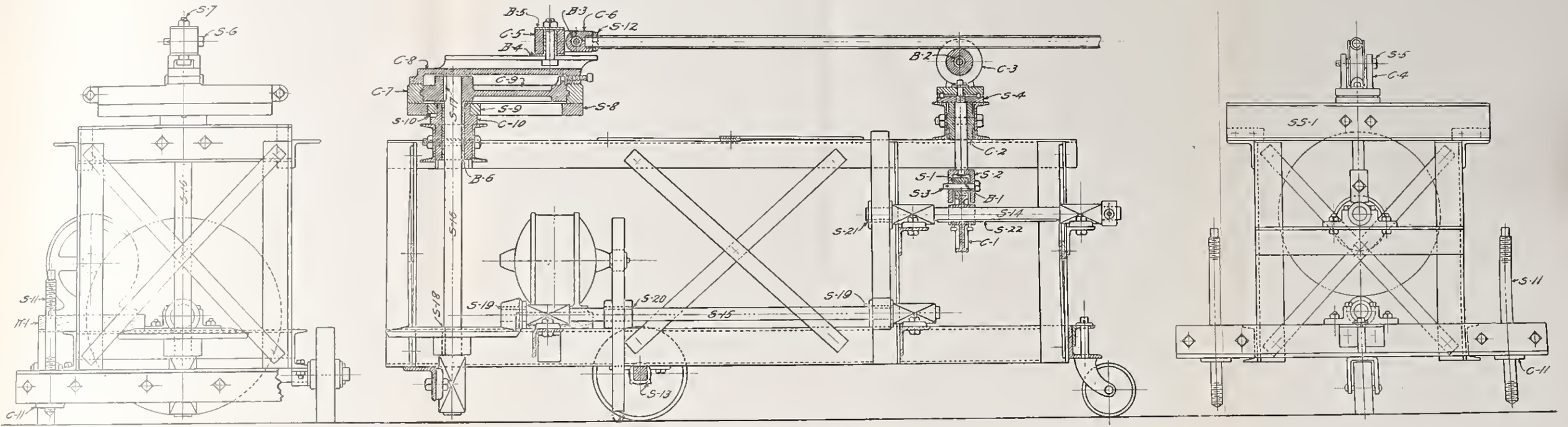


FIG. 6.—Glass stirring machine.

ring air into the melt and causing big bubbles. When the temperature of the glass has been reduced to the proper point, stirring ceases and the thimble is removed. To prevent striae from reforming, the metal is cooled as quickly as possible from this point to about 700° C. This is done by tipping the pot back, placing a fire brick support under it, and directing a stream of compressed air against the bottom for 3 hours. When the pot is cool enough to handle, it is removed from the furnace and the clay broken from the glass. The latter is then given a preliminary inspection and broken to suitable sizes for molding.

Molding and Annealing.—A great deal of good optical glass was ruined during molding and annealing. Several methods of molding were tried. In all methods the glass was gradually heated to the softening temperature in a furnace. The treatment following this in one method was to place the glass in clay trays which were coated with a mineral powder to prevent the glass from sticking to the tray. The tray was placed in a furnace sufficiently hot to melt the glass until it flowed. It was then turned out of the tray and pressed. Another method followed was to place the heated piece in a small furnace, the floor of which was covered with the mineral powder. As the glass softened it was paddled by hand into a block which would fit into the iron mold of a press. Several powders were tried in the above methods, such as alumina, flint, mica, kaolin, lime, etc. The latter method is known to be the most satisfactory of all from the standpoint of economy and quality, but on account of lack of skilled workmen, could not be used. It was found necessary to resort to the following method: The glass was "stuck up" on a punty and softened in a glory hole, shaped at a bench by paddling, and then sheared into a mold and pressed. Small lenses and prisms do not require shaping. After pressing, the glass is annealed according to schedules which depend upon the kind of glass. For all kinds the heating schedule was the same for the first 12 hours. The temperature was brought to 200° C the first 4 hours, to 300° in 8 hours, and to 400° at the twelfth hour. During the next $3^1/_2$ hours the final temperature was reached, depending upon the kind of glass, as follows: flint, 480°; light crown, 520°; boro

silicate, 550°; light barium crown, 610°; dense barium crown, 625°. The experimental work necessary in the determination of these temperatures was done by Dr. A. Q. Tool, of the Bureau of Standards. After reaching the maximum temperature, the glass was cooled at the rate of 5° per hour. No doubt all optical glass makers have endeavored to gather the glass directly from the pot and press it into lenses and prisms. This method would obviate the breaking of the pot and it could be used for several successive melts. The great waste of labor and glass incidental to breaking the glass to size and molding it would be eliminated, and a very substantial saving in fuel would be effected. In order to stir while gathering, the stirring rod is operated from the rear of the furnace, while the gathering is done from the front. The gatherer endeavors to follow directly in the path of the rod which insures the gathering of the most homogeneous glass. This work was carried on under the direction of Capt. H. C. Fry, Jr., who gave it considerable time and attention. The experiment was only partially successful, due to the gatherers not being accustomed to working with optical glass. Further experiments in gathering are contemplated and are thought necessary to conclusively demonstrate the success of the method as applied to commercial production. Decisive results could be obtained only by a comparison of several months' production by gathering compared with the old method.

Chemical Composition.—The compositions of the glasses made at the Bureau of Standards were developed to meet the demands of the military forces. Most of the types of glasses were based on analyses of glasses manufactured in Europe. Of course, some difficulty was experienced in converting the analytical results into a raw batch which would yield a glass of the desired optical constants. Experience demonstrated that the optical constants obtained by varying the chemical formula did not agree with the predicted result. It is true that occasionally the final result was close to the theoretical prediction, but in many cases it was wide of the mark. The optical properties do not depend entirely upon chemical composition. Other factors controlling them are temperature, time of melting, pressures, etc. Theoretically, the optical constants should not vary from melt to melt, pro-

viding all conditions are equal. In large scale production, however, the optical constants are not always the same for like compositions. This is illustrated in Table 3. From this table it is apparent that the optical results obtainable from a particular composition cannot be accurately judged from one melt. In fact, the indices will occasionally vary in the third decimal place in samples taken from different parts of the same melt.

TABLE 3.—VARIATION OF INDICES WITH SAME COMPOSITION AND OTHER CONDITIONS APPARENTLY EQUAL.

		N_D.	ν.
		1.6282	36.7
		1.6283	36.7
SiO_2	44.4	1.6268	36.7
PbO	46.0	1.6278	36.8
K_2O	4.1	1.6288	36.5
Na_2O	3.5	1.6248	36.6
CaO	2.0	1.6242	37.2
As_2O_3	0.4	1.6221	37.4
		1.6285	36.6
		1.6265	36.7

The effect of change in composition on index and dispersion is shown in Table 4. The indices herein presented are the averages of the indicated number of melts.

TABLE 4.—VARYING INDICES WITH COMPOSITION.

Designation.	Melts averaged.	SiO_2.	K_2O.	Na_2O	CaO.	ZnO.	B_2O_3.	PbO.	BaO.	As_2O_3.	N_D.	ν.
B–4..	10	44.4	5.0	3.5	3.0			44.0		0.2	1.62446	37.2
B–8..	10	45.6	4.1	3.5	3.0			44.0		0.2	1.62741	36.9
B–13.	10	44.4	4.1	3.5	2.0			46.0		0.4	1.62658	36.8
E–1..	7	48.1	7.5	1.0		10.1	4.5		28.3	0.4	1.56907	57.3
E–2..	7	48.0	6.1	2.0		10.0	4.0		29.5	1.4	1.57343	56.7
E–3..	7	47.6	6.0	2.0		9.8	4.0		30.5	1.5	1.57700	56.5
E–4..	5	46.0	6.1	2.0		10.5	5.5		29.5	1.5	1.57990	56.7

It should be noted that all the compositions given in this paper are the theoretical compositions used as a basis for making up the

TABLE 5.—MEDIUM FLINT.

Melt No.	Batch No.	% SiO_2.	% PbO.	% K_2O.	% Na_2O.	% CaO.	% B_2O_3.	% As_2O_3.	N_D.	N_C.	N_F.	N_G.	ν.	Transmission T.	Melting time.	Type of pot.	Per cent of cullet used.
21	B-1	44.1	44.7	9.8	1.2			0.2	1.60683	1.60222	1.61837	1.62808	37.5		57	Covered	20
54	B-2	44.4	44.9	6.1	1.2	3.2		0.2	1.62229	1.61751	1.63424	1.64438	37.2	98.3	52	Open	10
58	B-3	41.8	45.3	6.16	1.21	3.23	2.01		1.62890	1.62411	1.64095	1.65121	37.3	98.4	52	Open	10
75	B-4	44.4	44.0	5.0	3.5	3.0		0.2	1.62082	1.61762	1.63435	1.64447	37.2	99.1	49	Open	10
82	B-4	44.4	44.0	5.0	3.5	3.0		0.2	1.62241	1.61605	1.63267	1.64276	37.3	98.4	50	Open	0
89	B-4	44.4	44.0	5.0	3.5	3.0		0.2	1.62031	1.61559	1.63211	1.64216	37.5		51	Open	0
95	B-4	44.4	44.0	5.0	3.5	3.0		0.2	1.62441	1.61958	1.63648	1.64679	37.6	98.1	54	Open	0
99	B-4A	44.4	44.0	5.0	3.5	3.0		0.2	1.62659	1.62175	1.63872	1.64903	36.9	98.0	54	Open	10
100	B-4A	44.4	44.0	5.0	3.5	3.0		0.2	1.62683	1.62201	1.63905	1.64937	36.8	98.2	52	Open	10
109	B-4A	44.4	44.0	5.0	3.5	3.0		0.2	1.62781	1.62291	1.63997	1.65035	36.8	98.2	52	Open	10
110	B-4A	44.4	44.0	5.0	3.5	3.0		0.2	1.62495	1.62013	1.63701	1.64747	37.4	97.6	52	Open	10
111	B-4A	44.4	44.0	5.0	3.5	3.0		0.2	1.62528	1.62047	1.63728	1.64755	37.2	98.0	52	Open	10
112	B-4A	44.4	44.0	5.0	3.5	3.0		0.2	1.62518	1.62034	1.63723	1.64749	37.0	97.5	52	Open	10
128	B-5	44.5	42.0	7.0	3.5	3.0		0.2	1.60899	1.60450	1.62014	1.62959	38.9		49	Open	10
130	B-5A	44.5	42.0	7.0	3.5	3.0		0.2	1.59694	1.59270	1.60747	1.61636	38.4	98.5	49	Open	10
141	B-6	46.5	42.0	5.0	3.5	3.0		2.0	1.61811	1.6132	1.6294	1.63693	38.0	97.9	54	Open	10
142	B-7	45.6	43.2	4.9	3.43	2.95		0.2	1.62841	1.62365	1.64073	1.65115	26.8	98.7	54	Open	10
143	B-7	45.6	43.2	4.9	3.43	2.95		0.2	1.62426	1.61947	1.63626		37.2	98.2	55	Open	10
144	B-8	45.6	44.0	4.1	3.43	2.95		0.2	1.62546	1.62064	1.63753	1.64774	37.0	98.3	52	Open	10
158	B-8	45.6	44.0	4.1	3.43	2.95		0.2	1.62769	1.6227	1.6397		36.9	98.6	55	Open	25
160	B-8	45.6	44 0	4.1	3.43	2.95		0.2	1.6248	1.6200	1.6367		37.3	98.9	56	Open	25
161	B-8	45.6	44.0	4.1	3.43	2.95		0.2	1.6283	1.6234	1.6405		37.1	97.2	57	Open	10
162	B-8*a*	45.6	44.0	4.1	3.43	2.95		0.2	1.62684	1.62202	1.63892		37.1	98.3	55	Open	25
163	B-8*a*	45.6	44.0	4.1	3.43	2.95		0.2	1.6272	1.6223	1.6393		37.1	98.3	56	Open	25
164	B-8*a*	45.6	44.0	4.1	3.43	2.95		0.2	1.6283	1.6236	1.6408		46.8	97.9	58	Open	25
165	B-8*a*	45.6	44.0	4.1	3.43	2.95		0.2	1.62655	1.6216	1.6588		36.3	98.6	58	Open	25
166	B-8*a*	45.6	44.0	4.1	3.43	2.95		0.2	1.63117	1.6263	1.6435		36.5	97.8	56	Open	25
167	B-8*a*	45.6	44.0	4.1	3.43	2.95		0.2	1.6277	1.6229	1.6398		37.1	98.3	56	Open	25
168	B-8*a*	45.6	44.0	4.1	3.43	2.95		0.2	1.6250	1.6202	1.6369		37.4	98.3	56	Open	25
176	B-9	44.16	47.50	7.70			0.52	0.2	1.6243	1.6195	1.6366		36.4	98.5	56	Open	10

TABLE 5.—MEDIUM FLINT (*Continued*).

182	B-10	44.16	47.50	5.70	2.0		0.52	0.2	1.6237	1.6188	1.6360		36.3	98.7	53	Open	10
185	B-10	44.16	47.50	5.70	2.0		0.52	0.2	1.6222	1.6173	1.6344		36.4	98.9	49	Open	10
192	B-11	46.6	44.0	4.1	3.5	1.8	0.52	0.2	1.6141	1.6095	1.6257		37.9	98.1	55	Open	10
193	B-11	46.6	44.0	4.1	3.5	1.8	0.52	0.2	1.6203	1.6156	1.6321		37.5	98.1	55	Open	0
194	B-11*a*	46.6	44.0	4.1	3.5	1.8	0.52	0.2	1.6162	1.6118	1.6281		37.8		55	Open	10
209	B-12	45.6	45.0	4.1	3.5	1.8		0.4	1.6192	1.6145	1.6311		37.2	98.5	56	Open	10
211	B-12	45.6	45.0	4.1	3.5	1.8		0.4	1.6242	1.6194	1.6363		37.0	98.6	52	Open	20
214	B-13	44.4	46.0	4.1	3.5	2.0		0.4	1.6243	1.6194	1.6364		36.8		52	Open	25
215	B-13*a*	44.4	46.0	4.1	3.5	2.0		0.4	1.6274	1.6225	1.6397		36.4		50	Open	20
219	B-13*a*	44.4	46.0	4.1	3.5	2.0		0.4	1.6273	1.6224	1.6397		36.2		54	Open	30
220	B-13*a*	44.4	46.0	4.1	3.5	2.0		0.4	1.6315	1.6266	1.6441		36.2		55	Open	30
226	B-13*b*	44.4	46.0	4.1	3.5	2.0		0.4	1,6268	1.6219	1.6391		36.5	97.5	48	Open	50
254	B-13*c*	44.4	46.0	4.1	3.5	2.0		0.4	1.6165	1.6119	1.6282		37.7		30	Open	40
255	B-13*c*	44.4	46.0	4.1	3.5	2.0		0.4	1.6215	1.6167	1.6333		37.3	98.4	33	Open	40
256	B-13*c*	44.4	46.0	4.1	3.5	2.0		0.4	1.6341	1.6291	1.6469		35.8	98.9	34	Covered	0
258	B-13*c*	44.4	46.0	4.1	3.5	2.0		0.4	1.6282	1.6235	1.6407		36.7	98.6	30	Covered	40
259	B-13*c*	44.4	46.0	4.1	3.5	2.0		0.4	1.6283	1.6234	1.6406		36.7	98.4	32	Open	40
260	B-13*c*	44.4	46.0	4.1	3.5	2.0		0.4	1.6268	1.6219	1.6390		36.7	98.5	32	Open	40
262	B-13*c*	44.4	46.0	4.1	3.5	2.0		0.4	1.6278	1.6228	1.6399		36.8	98.3	32	Open	40
263	B-13*c*	44.4	46.0	4.1	3.5	2.0		0.4	1.6288	1.6239	1.6411		36.5		32	Open	40
265	B-13*c*	44.4	46.0	4.1	3.5	2.0		0.4	1.6248	1.6198	1.6369		36.6	98.4	33	Open	45
266	B-13*c*	44.4	46.0	4.1	3.5	2.0		0.4	1.6242	1.6194	1.6362		37.2	98.9	32	Open	40
267	B-13*c*	44.4	46.0	4.1	3.5	2.0		0.4	1.6221	1.6174	1.6340		37.4	98.6	32	Open	40
268	B-13*c*	44.4	46.0	4.1	3.5	2.0		0.4	1.6285	1.6236	1.6408		36.6	98.1	34	Open	40
269	B-13*c*	44.4	46.0	4.1	3.5	2.0		0.4	1.6263	1.6215	1.6385		36.7	98.5	32	Open	40
270	B-13*c*	44.4	46.0	4.1	3.5	2.0		0.4	1.6272	1.6224	1.6394		36.9	98.3	31	Open	40
271	B-13*c*	44.4	46.0	4.1	3.5	2.0		0.4	1.6266	1.6217	1.6388		36.6	98.5	30	Open	40
272	B-13*c*	44.4	46.0	4.1	3.5	2.0		0.4	1.6231	1.6181	1.6350		36.8	98.9	31	Open	40
275	B-13*c*	44.4	46.0	4.1	3.5	2.0		0.4	1.6300	1.6250	1.6423		36.3	98.7	33	Covered	40
276	B-13*c*	44.4	46.0	4.1	3.5	2.0		0.4	1.6242	1.6194	1.6363		37.0	98.6	31	Open	40
277	B-15	44.9	45.5	4.1	3.5	2.0		0.4	1.6233	1.6233	1.6185		36.7	98.4	33	Open	40
280	B-16	45.4	45.0	4.1	3.5	2.0		0.4	1.6193	1.6146	1.6312		37.2	99.1	31	Open	40
285	B-16	45.4	45.0	4.1	3.5	2.0	BaO	0.4	1.6211	1.6163	1.6330		37.1	98.4	31	Open	40
287	B-17	46.4	44.0	4.1	3.5	2.0	1.0	0.4	1.6218	1.6170	1.6338		37.0	98.5	30	Open	40
288	B-17	46.4	44.0	4.1	3.5	2.0	1.0	0.4	1.6173	1.6126	1.6291		37.3	98.5	31	Open	40
289	B-17	46.4	44.0	4.1	3.5	2.0	1.0	0.4	1.6200	1.6153	1.6319		37.2	98.6	31	Covered	40

TABLE 6.—LIGHT FLINT.

Melt. No.	Batch No.	% SiO_2.	% PbO.	% K_2O.	% Na_2O.	% CaO.	% As_2O_3.	N_D.	N_C.	N_F.	N_G.	ν.	Transmission. T.	Melting time.	Type of pot.	Per cent of cullet used.
29	A-2	53.9	36.7	6.0	1.0	1.96	0.3	1.57988	1.57583	1.58983	1.59816	41.6	99.5	54	Open	17
33	A-2	53.9	36.7	6.0	1.0	1.96	0.6	1.58484	1.58070	1.59509	1.60366	40.5	99.3	54	Open	15
52	A-2	53.9	36.7	6.0	1.0	1.96	0.4	1.59138	1.58726	1.60159	1.60998	41.3	99.0	53	Open	10
56	A-2	53.9	36.7	6.0	1.0	1.96	0.3	1.5803	1.5762	1.5902	1.5986	41.0	99.1	55	Open	10
188	A-4	53.9	35.2	7.64	1.0	1.96	0.3	1.5805	1.5765	1.5903		41.9	99.3	56	Open	0

TABLE 7.—DENSE FLINT.

Melt. No.	Batch No.	% SiO_2.	% PbO.	% K_2O.	% CaO.	% Na_2O.	% Sb_2O_3.	% As_2O_3.	N_D.	N_C.	N_F.	N_G.	ν.	Transmission. T.	Melting time.	Type of pot.	Per cent of cullet used.
65	C-2	39.9	48.8	6.8	4.8			0.2	1.64799	1.64272	1.66125	1.67259	35.0	99.1	51	Open	0
76	C-3	39.0	49.0	4.0	4.0	3.0	1.0	0.2	1.65555	1.65019	1.66922	1.68091	34.4	98.9	54	Covered	60

TABLE 8.—BARIUM FLINT.

Melt. No.	Batch No.	% SiO_2.	% PbO.	% K_2O.	% Na_2O.	% ZnO.	% B_2O_3.	% BaO.	% CaO.	% As_2O_3.	N_D.	N_C.	N_F.	N_G.	ν	Transmission. T.	Melting time.	Type of pot.	Per cent of cullet used.
135	H-2	53.69	16.71	8.31	1.69	2.53	2.68	14.27		0	1.56823	1.5648	1.5764		48.6	98.6	54	Open	10
145	H-3	58.69	12.71	8.31	1.69	2.53	1.68	14.27		0.4	1.5521	1.5489	1.5597	1.56630	51.0	99.0	50	Open	25
155	H-3*a*	58.69	12.71	8.31	1.69	2.53	1.68	14.27		0.6	1.5496	1.5465	1.5571		51.9	99.3	53	Open	10
156	H-3*a*	58.69	12.71	8.31	1.69	2.53	1.68	14.27		0.5	1.5567	1.5535	1.5644		51.0	99.4	53	Open	0
169	H-3*b*	58.69	12.71	8.31	1.69	2.53	1.68	14.27		0.6	1.5582	1.5551	1.5660		51.2	99.5	52	Open	25
190	H-3*c*	58.69	12.71	8.31	1.69	2.53	1.68	14.27		0.4	1.5529	1.5498	1.5603		52.6	99.6	85	Open	10
199	H-3*d*	58.69	12.71	8.31	1.69	2.53	1.68	14.27		0.3	1.5542	1.5413	1.5510		56.3	99.3	50	Open	10
200	H-3*d*	58.69	12.71	8.31	1.69	2.53	1.68	14.27		0.3	1.5484	1.5454	1.5557		53.3	99.8	52	Open	10
205	H-3*e*	58.69	12.71	8.31	1.69	2.53	1.68	14.27		0.4	1.5503	1.5474	1.5579		52.5	99.3	50	Open	30
207	H-3*e*	58.69	12.71	8.31	1.69	2.53	1.68	14.27		0.4	1.5493	1.5462	1.5566		53.0	99.7	74	Open	30
208	H-3*e*	58.69	12.71	8.31	1.69	2.53	1.68	14.27		0.4	1.5521	1.5491	1.5596		52.3		51	Open	0
217	H-3*f*	58.81	12.71	8.31	1.69	2.53	1.68	14.27	1.00	0.5	1.5488	1.5457	1.5561		52.7		51	Open	33
218	H-3*f*	58.81	12.71	8.31	1.69	2.53	1.68	12.27	1.00	0.5	1.5500	1.5470	1.5575		52.6		53	Open	33
223	H-4	58.8	13.71	8.31	1.7	2.53	1.68	12.27	1.00	0	1.5514	1.5483	1.5589		52.1		45	Open	30
224	H-4	58.8	13.71	8.31	1.7	2.53	1.68	12.27	1.00	0	1.5518	1.5487	1.5593		52.1		48	Open	25
245	H-4*a*	58.8	13.71	8.31	1.7	2.53	1.68	12.27	1.00	0.4	1.5545	1.5514	1.5620		52.3	99.2	38	Open	30
253	H-4*a*	58.8	13.71	8.31	1.7	2.53	1.68	12.27	1.00	0.4	1.5537	1.5506	1.5612		52.2	99.3	27	Covered	75
261	H-4	58.81	13.71	8.31	1.69	2.53	1.68	12.27	1.00		1.5544	1.5513	1.5618		52.6	99.0	31	Open	80
264	H-4	58.81	13.71	8.31	1.69	2.53	1.68	12.27	1.00		1.5531	1.5500	1.5606		51.7	99.1	32	Open	100

TABLE 9.—LIGHT CROWN.

Melt. No.	Batch No.	% SiO_2.	% B_2O_3.	% K_2O.	% Na_2O.	% BaO.	% ZnO.	% As_2O_3.	N_D.	N_C.	N_F.	N_G.	ν.	Transmission, T.	Melting time.	Type of pot.	Per cent of cullet used.
20	D-1	68.5	3.5	5.0	12.0	9.7	1.0	0.2	1.5179	1.5153	1.5238	1.5286	60.9	99.0	60	Open	15
36	D-1	68.5	3.5	5.0	12.0	9.7	1.0	0.5	1.5151	1.5140	1.5226	1.5274	60.0	99.6	53	Open	15
41	D-1	68.5	3.5	5.0	12.0	9.7	1.0	0.5	1.5153	1.5128	1.5213	1.5261	60.5	98.9	50	Open	15
42	D-1	68.5	3.5	5.0	12.0	9.7	1.0	0.5	1.51656	1.51416	1.52255	1.52734	61.3	98.7	50	Open	5
43	D-1	68.5	3.5	5.0	12.0	9.7	1.0	0.5	1.51717	1.51462	1.52317	1.52791	62.5	98.6	50	Open	15
46	D-1	68.5	3.5	5.0	12.0	9.7	1.0	0.5	1.5165	1.5140	1.5225	1.5276	60.3	98.6	53	Open	15
49	D-1	68.5	3.5	5.0	12.0	9.7	1.0	0.2	1.52905	1.52638	1.53520	1.54006	60.0	98.9	53	Open	10
55	D-1	68.5	3.5	5.0	12.0	9.7	1.0	0.2	1.5179	1.5153	1.5239	1.5288	60.0	99.1	51	Open	10
71	D-1	68.5	3.5	5.0	12.0	9.7	1.0	0.4	1.51554	1.51297	1.52151	1.52633	60.4	98.5	49	Open	10
78	D-2	68.5	3.0	5.0	11.0	10.6	1.5	0.4	1.5155	1.51296	1.52157	1.52647	59.9	98.8	51	Open	0
80	D-2	68.5	3.0	5.0	11.0	10.6	1.5	0.4	1.5158	1.51326	1.52189	1.52680	59.8	99.1	53	Open	10
90	D-2	68.5	3.0	5.0	11.0	10.6	1.5	0.4	1.5160	1.51347	1.52207	1.62697	60.0	98.6	44	Open	10
91	D-2	68.5	3.0	5.0	11.0	10.6	1.5	0.4	1.51475	1.51223	1.52076	1.52556	60.2	98.8	51	Open	10
103	D-3	67.0	3.5	5.0	12.0	10.6	1.5	0.4	1.5175	1.51455	1.52319	1.52803	59.9	98.7	54	Open	10
113	D-3	67.0	3.5	5.0	12.0	10.6	1.5	0.4	1.51771	1.51528	1.52394		59.7	98.8	52	Open	40
123	D-3*b*	67.0	3.5	5.0	12.0	10.6	1.5	0.4	1.51708	1.5147	1.5234	1.52819	59.6	98.9	50	Open	40

TABLE 10.—BOROSILICATE.

Melt. No.	Batch No.	% SiO_2.	% B_2O_3.	% K_2O.	% Na_2O.	% CaO.	% BaO.	% ZnO.	% As_2O_3.	N_D.	N_C.	N_F	N_G.	ν.	Transmission. T.	Melting time.	Type of pot.	Per cent of cullet used.
40	G-2	69.5	8.9	8.39	8.2		3.01	0.25	0.31	1.51193	1.50949	1.51761	1.52210	63.1	98.8	50	Open	13
44	G-2	69.5	8.9	8.39	8.2		3.01	0.25	0.31	1.51254	1.51012	1.51816	1.52260	63.8	99.0	51	Open	15
47	G-2	69.5	8.9	8.39	8.2		3.01	0.25	0.31	1.5101	1.5077	1.5157	1.5200	64.0	98.3	51	Open	15
72	G-3	68.0	6.0	8.0	10.0		8.0	2.0	0.2	1.51718	1.51467	1.52307	1.53778	61.5	99.0	54	Open	10
83	G-3	68.0	6.0	6.0	10.0		8.0	2.0	0.2	1.51640	1.51390	1.52229	1.52701	61.6	97.8	76	Open	0
86	G-4	68.0	8.0	6.0	10.0		8.0	2.0	0.2	1.51966	1.51716	1.52555	1.53029	61.9	99.1	48	Open	0
92	G-4	68.0	8.0	6.0	10.0		8.0	2.0	0.2	1.52253	1.52015	1.52845	1.53321	63.0	99.0	58	Open	0
94	G-5	68.0	8.0	6.0	10.0		8.0	2.85	0.2	1.51997	1.51742	1.52584	1.53058	61.8	98.8	51	Open	30
187	G-6	64.2	11.0	8.27	9.45		7.08	2.85	0.2	1.5243	1.5219	1.5302		63.3	98.9	58	Open	0
201	G-7	64.2	13.0	8.27	9.45		5.08	2.85	0.2	1.5220	1.5196	1.5277		64.2	99.7	56	Open	10
210	G-8	64.2	11.0	8.27	9.45	1.0	6.08	2.85	0.2	1.5240	1.5216	1.5298		63.4	99.1	50	Open	40
240	G-9	64.2	12.5	8.27	9.45	1.0	4.58	2.85	0.4	1.5245	1.5221	1.5303		64.0	98.8	35	Covered	60
241	G-9	64.2	12.5	8.27	9.45	1.0	4.58	2.85	0.4	1.5252	1.5241	1.5303		64.4	99.0	38	Covered	75

TABLE 11.—LIGHT BARIUM CROWN.

Melt. No.	Batch No.	% SiO_2.	% B_2O_3.	% K_2O.	% Na_2O.	% ZnO.	% BaO.	% CaO.	% As_2O_3.	N_D.	N_C.	N_F.	N_G.	ν.	Transmission, T.	Melting time.	Type of pot.	Per cent of cullet used.
27	E-1	48.1	4.5	7.5	1.0	10.1	28.3	2.0	0.4	1.5615	1.55682	1.56828	1.57367	58.1	98.0	52	Open	15
30	E-1	48.1	4.5	7.5	1.0	10.1	28.3		0.4	1.57038	1.56740	1.57740	1.58299	57.0	95.0	56	Open	15
50	E-1	48.1	4.5	7.5	1.0	10.1	28.3		0.4	1.5701	1.5669	1.5768	1.5827	57.6	98.3	56	Open	10
51	E-1	48.1	4.5	7.5	1.0	10.1	28.3		0.4	1.56918	1.5663	1.5762	1.5818	57.3	98.9	52	Open	10
57	E-1	48.1	4.5	7.5	1.0	10.1	28.3		0.4	1.5698	1.5680	1.5779	1.5835	57.4	97.4	50	Open	10
59	E-1	48.1	4.5	7.5	1.0	10.1	28.3	2.0	0.4	1.57202	1.56908	1.57907	1.58479	57.3	97.9	52	Open	10
66	E-1	48.1	4.5	7.5	1.0	10.1	28.3		0.4	1.57050	1.56758	1.57763	1.58321	56.8	97.8	51	Open	10
87	E-2	48.0	4.0	6.1	2.0	10.0	29.5		1.4	1.57340	1.57043	1.58057	1.58625	56.7	98.7	52	Open	0
105	E-1	48.0	4.5	6.1	2.0	10.0	29.5		1.4	1.57542	1.57253	1.58272	1.58855	57.5	98.3	52	Open	50
115	E-2*a*	48.0	4.0	6.1	2.0	10.0	29.5		1.4	1.57420	1.57126	1.58139	1.58720	56.7	97.5	49	Open	50
116	E-2*a*	48.0	4.0	6.1	2.0	10.0	29.5		1.4	1.57409	1.57112	1.58125	1.58703	56.6	98.6	51	Open	50
117	E-2*a*	48.0	4.0	6.1	2.0	10.0	29.5		1.4	1.57427	1.57122	1.58133		56.8	98.1	50	Open	10
121	E-2*a*	48.0	4.0	6.1	2.0	10.0	29.5		1.4	1.56752	1.56468	1.57465	1.58036	57.0	99.5	48	Open	10
125	E-2B	48.0	4.0	6.1	2.0	10.0	29.5		1.4	1.57511	1.57213	1.58229	1.58813	56.6	98.7	50	Open	10
126	E-3	47.6	3.95	6.0	1.98	9.8	30.5	2.0	1.5	1.57508	1.57207	1.58225	1.58806	56.3	98.8	52	Open	10
133	E-3	47.6	3.95	6.0	1.98	9.8	30.5		1.5	1.57850	1.57547	1.58580	1.59123	56.0	98.7	51	Open	10
136	E-3	47.6	3.95	6.0	1.98	9.8	30.5		0.0	1.57712	1.57413	1.58432	1.59916	56.7	97.8	53	Open	10
138	E-3	47.6	3.95	6.0	1.98	9.8	30.5		0.0	1.5759	1.57271	1.58284		56.6	98.3	51	Open	10
139	E-3	47.6	3.95	6.0	1.98	9.8	30.5		0.0	1.5765	1.57354	1.58369		56.8	98.3	58	Open	10
140	E-3	47.6	3.95	6.0	1.98	9.8	30.5		0.0	1.58045	1.57748	1.58781		56.4	97.9	56	Open	10
148	E-3*a*	47.6	3.95	6.0	1.98	9.8	30.5		0.6	1.5755	1.57237	1.58253		56.5	98.4	55	Open	25
149	E-3*a*	47.6	3.95	6.0	1.98	9.8	30.5		0.5	1.5995	1.59616	1.50714	1.61348	54.6	98.1	55	Open	25
150	E-3*b*	47.6	3.95	6.0	1.98	9.8	30.5		0.5	1.5789	1.57623	1.58652	1.59243	56.5	99.1	54	Open	25
152	E-4	46.0	5.5	6.1	2.0	10.5	29.5		0.5	1.5790	1.5760	1.5862		56.5	98.5	55	Open	10
153	E-4	46.0	5.5	6.1	2.0	10.5	29.5		0.5	1.5771	1.5742	1.5842		56.7	98.4	55	Open	10
154	E-4	46.0	5.5	6.1	2.0	10.5	29.5		0.5	1.5777	1.5747	1.5848	1.59075	57.1	98.7	55	Open	25
172	E-4	46.0	5.5	6.1	2.0	10.5	29.5		0.5	1.5826	1.5796	1.5899		56.7	97.8	60	Open	25
173	E-4*a*	46.0	5.5	6.1	2.0	10.5	29.5		0.5	1.5831	1.5801	1.5904		56.6	98.5	55	Open	25
175	E-5	46.3	6.5	7.2	2.0	8.5	29.5		0.5	1.5837	1.5796	1.5899		56.4	97.5	55	Open	10
178	E-6	48.1	4.57	8.0	2.0	10.4	26.5		0.4	1.5741	1.5711	1.5812		56.5	98.3	59	Open	10

TABLE 11—LIGHT BARIUM CROWN (*Continued*).

195	E-6	48.1	4.57	8.0	2.0	10.4	26.5		0.4	1.5693	1.5664	1.5763		57.6	98.0	58	Open	10
196	E-5*a*	46.3	6.5	7.2	2.0	8.5	29.5		0.4	1.5766	1.5737	1.5837		57.6	96.8	55	Open	10
229	E-7	48.1	4.6	8.0	2.0	8.4	26.5	2.0	0.5	1.5755	1.5726	1.5826		57.2	98.5	39	Open	50
231	E-7	48.1	4.6	8.0	2.0	8.4	26.5	2.0	0.5	1.5724	1.5695	1.5795		56.9	98.5	44	Covered	50
232	E-7	48.1	4.6	8.0	2.0	8.4	26.5	2.0	0.5	1.5750	1.5720	1.5820		57.7	98.2	44	Covered	50
233	E-7	48.1	4.6	8.0	2.0	8.4	26.5	2.0	0.5	1.5723	1.5694	1.5793		57.9	98.8	44	Covered	50
234	E-7	48.1	4.6	8.0	2.0	8.4	26.5	2.0	0.5	1.5705	1.5676	1.5774		58.5	98.8	34	Covered	50
236	E-7	48.1	4.6	8.0	2.0	8.4	26.5	2.0	0.5	1.5753	1.5724	1.5824		57.5	98.1	36	Covered	50
242	E-7	48.1	4.6	8.0	2.0	8.4	26.5	2.0	0.5	1.5728	1.5699	1.5798		57.7	98.8	35	Covered	50
243	E-7	48.1	4.6	8.0	2.0	8.4	26.5	2.0	0.5	1.5737	1.5706	1.5806		57.5	99.2	33	Open	10
237	E-7	48.1	4.6	8.0	2.0	8.4	26.5	2.0	0.5	1.5725	1.5696	1.5795		57.9	98.6	35	Covered	50
238	E-7	48.1	4.57	8.0	2.0	8.4	26.5	2.0	0.5	1.5764	1.5734	1.5835		57.1	98.7	36	Covered	50
244	E-7	48.1	4.57	8.0	2.0	8.4	26.5	2.0	0.5	1.5717	1.5688	1.5787		57.5	99.0	34	Open	10
250	E-7	48.1	4.57	8.0	2.0	8.4	26.5	2.0	0.5	1.5723	1.5694	1.5794		57.2	99.0	35	Covered	60
252	E-7	48.1	4.57	8.0	2.0	8.4	26.5	2.0	0.5	1.5701	1.5671	1.5772		56.4	98.8	35	Covered	60
257	E-7	48.1	4.57	8.0	2.0	8.4	26.5	2.0	0.5	1.5721	1.5692	1.5791		57.8	98.8	38	Covered	60

Table 12.—DENSE BARIUM CROWN.

Melt. No.	Batch No.	% SiO_2.	% B_2O_3.	% K_2O.	% CaO.	% ZnO.	% PbO.	% BaO.	% Al_2O_3.	% As_2O_3.	N_D.	N_C.	N_F.	N_G.	ν.	Transmission, T.	Melting time.	Type of pot.	Per cent of cullet used.
53	F-1	39.6	5.0		2.0	7.7	3.0	44.0		0.4	1.59051	1.58736	1.59773	1.60358	57.0	88.1	57	Open	10
122	F-2*b*	37.0	5.0	2.6	2.0	7.7	3.0	44.0		0.7	1.62146	1.61809	1.62968	1.63636	53.6	91.0	51	Open	10
124	F-2*b*	37.0	5.0	2.6		7.7	3.0	44.0		0.7	1.6199	1.61779	1.62939	1.63605	53.5	96.5	50	Open	10
127	F-2*b*	37.0	5.0	2.6		7.7	3.0	44.0		0.7	1.6177	1.6143	1.6258		53.9	96.6	51	Open	10
132	F-2*c*	37.0	5.0	2.6		7.7	3.0	44.0		0.7	1.6242	1.6208	1.6375		53.5	96.8	52	Open	10
151	F-3	37.0	5.0	2.6		7.7		47.0		0.7	1.6177	1.6144	1.6256		55.2	95.4	55	Open	25
171	F-4*a*	33.5	10.5	2.6		7.5		42.5	6.0	0.7	1.6040	1.6009	1.6115		57.1	91.1	63	Open	10
186	F-6	39.0	5.0	2.6	2.0	7.0		45.0	1.0	0.4	1.6098	1.60658	1.61749		55.9	96.0	62	Open	10
191	F-6*a*	39.0	5.0			7.0		45.0	1.0	0.4	1.6046	1.6015	1.6121		57.0	97.2	56	Open	10
216	F-9	34.5	9.1		2.0	7.8		42.0	4.0	0.6	1.6055	1.6024	1.6131		56.8	97.5	37	Open	10

batch weights and hence are not analyses of the finished glasses. This is due partly to undetermined changes in the raw materials as they lie in storage and partly to volatilization of materials in melting the glass.

The compositions used and indices obtained at the Bureau of Standards' factory are given in Tables 5 to 12. The optical properties were determined at the Bureau of Standards, Washington, D. C. In these tables the melting time in hours is shown, also the type of pot and the per cent of cullet used. All the data given in these tables were obtained from melts approximating 1000 pounds of glass.

Bureau of Standards,
Pittsburgh, Pa.

THE EFFECT OF ELECTROLYTES ON THE PROPERTIES OF GRAPHITE CRUCIBLE BODIES.[1]

By H. G. Schurecht.

Outline of Investigation.

The effect of electrolytes on the properties of graphite crucible bodies was studied by adding the electrolyte to each body in three different ways:

1. By pugging the body to stiff-mud consistency and then adding the electrolyte.

2. By wet-grinding the flake graphite with electrolyte and water in a wet pan, and then mixing the graphite thus treated with the clay and other constituents of the body.

3. By preparing slips with graphite and clay and then adding the electrolyte.

The following bodies were studied:

Table 1.—Composition of Bodies.

Number	1	2	3	4	5	6	7	8
	%	%	%	%	%	%	%	%
Alabama flake graphite	55	55	..	..	55	..	..	55
Alabama coked graphite	..	..	55	..	..	..	55	..
Alabama deflocculated graphite	..	..	..	55	..	..	..	..
Ceylon graphite	..	..	..	..	..	55	..	..
Dorset clay	35	..	..	..	45	45	45	..
Mississippi clay	..	35	35	35	..	..	..	45
Sand	5	5	5	5	..	..	..	..
Kaolin	5	5	5	5	..	..	..	..

The electrolytes used were NaOH and HCl. To study their effect on bodies in the plastic state the following properties were determined:

1. Working properties as judged by feel and behavior in jiggering crucibles.

[1] By permission of the Director, U. S. Bureau of Mines.

2. Dry transverse strength.
3. Dry density.
4. Dry porosity.
5. Drying volume shrinkage.

To determine the effect of NaOH on the viscosity of Bodies Nos. 5, 6, 7 and 8, the graphite was screened through a 40-mesh sieve and slips were prepared containing three parts solid and five parts water with varying percentages of NaOH. After standing twenty-four hours the slips were tested with the flow-viscosimeter, using an orifice of one-eighth inch. The viscosities were calculated by the use of the following formula:

$$V = S \times \frac{T_s}{T_w} \text{ in which}$$

V = Viscosity of slip.
S = Specific gravity of slip.
T_s = Time required for 250 cc. of slip at 18° C to flow through the orifice
T_w = Time required for 250 cc. of water at 18° C to flow through the orifice.

Results of the Investigation.

Working Properties.—When NaOH was added to Body No. 1, containing Dorset clay, it lost considerable plasticity. Attempts to make crucibles from this body, when NaOH was used, proved unsuccessful. On the other hand, the addition of HCl developed a body with better working properties than when untreated.

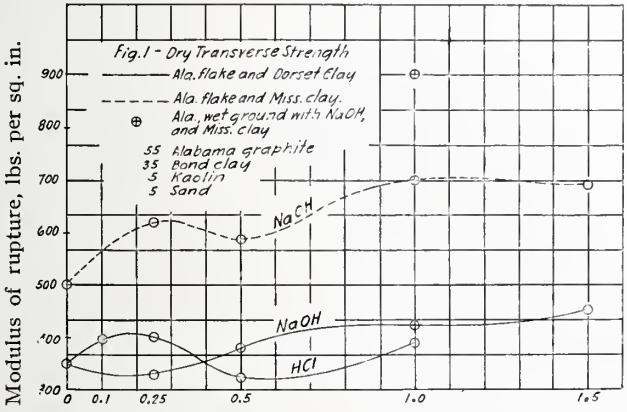
Adding NaOH to bodies containing Mississippi clay improved the working properties—its effect on this clay being the opposite of that on the Dorset clay.

Body No. 2 (containing no added electrolyte), with 55 per cent Alabama flake graphite and 35 per cent Mississippi bond clay was molded with difficulty—the body tearing badly from the jigger-tool and showing many laminations on the outside surface of the crucible. These difficulties were largely overcome by the use of one per cent of NaOH (in terms of clay).

In Body No. 4 the flake graphite was treated by adding one per cent NaOH and grinding in a wet pan for one and one-half

hours. Graphite thus treated developed considerable plasticity, had mechanical strength when dry, and when used to replace flake graphite produced a body whose jiggering behavior was much better than that of the body containing untreated flake graphite.

Dry Transverse Strength.—Adding NaOH to bodies containing Dorset clay at first decreased and, with larger additions, increased the dry strength of the body (see Fig. 1). This effect of



Per cent electrolyte in terms of clay.

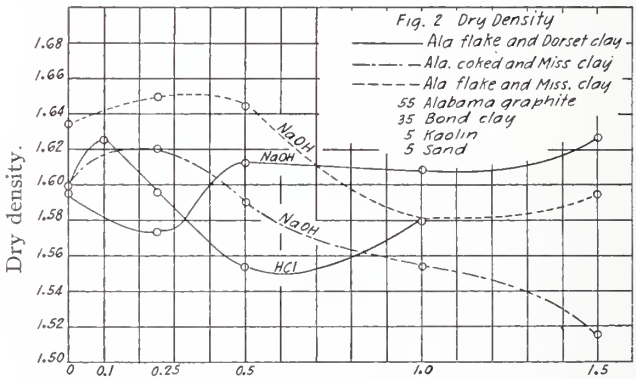
alkalies was probably due to flocculation of the clay with the smaller additions and to deflocculation with larger additions. As the grains in flocculated clay are more or less aggregates of many fine particles, we would expect this body to be mechanically weak. The deflocculated clay is extremely fine grained and, therefore, it would seem that a denser body can be molded and the mechanical strength thus increased. The effect of acids is the opposite of that produced by alkalies so it appears that the changes produced are functions of the alkalinity or acidity of the bodies.

When alkalies were added to bodies containing Mississippi clay the strength was increased. The effect of alkalies, therefore, seems to vary with different clays. This may be due to a difference in the initial alkalinity or acidity of the clays.

When the graphite, treated by pugging with NaOH, was added

to the body in the place of flake graphite, the modulus of rupture was 900 pounds per square inch. As the body containing flake graphite had a maximum strength of 700 pounds per square inch when treated with NaOH, it appears that there are certain advantages, as far as strength is concerned, gained by first pugging the graphite with NaOH before mixing with the clay. This increase in strength was partly due to the finer grains caused by wet grinding, but may also have been due to colloidal graphite produced by grinding.[1]

Density.—When NaOH in varying percentages was added to the body containing Dorset clay, its effect was to decrease the density when a small percentage (0.25 per cent in terms of clay) was used and then increase the density when larger percentages were used. HCl acted oppositely, in increasing the density with small additions and decreasing the density with the larger additions (see Fig. 2).



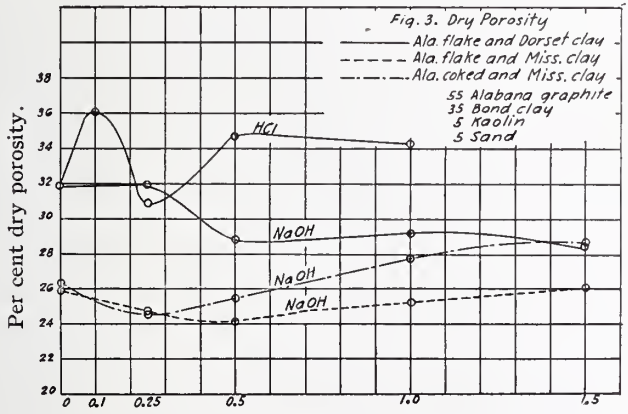
Per cent electrolyte in terms of clay.

The effect of NaOH on bodies containing Mississippi clay was similar to the effect of HCl on bodies containing Dorset clay.

Porosity.—Small percentages of HCl made the body containing Dorset clay more open while the addition of a larger per-

[1] E. G. Acheson, Brit. 111,434, Nov. 28, 1917, "Method of Deflocculating Solid Materials."

centage produced a less porous body (see Fig. 3). The alkali, on the other hand, made the body "tighter."



Per cent electrolyte in terms of clay.

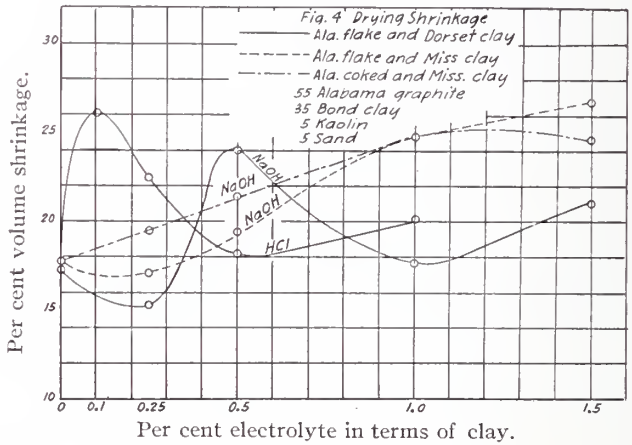
The effect of NaOH on bodies containing Alabama flake and coked graphites with Mississippi clay was to decrease the porosity when small percentages were used and to increase the porosity when larger percentages were used.

Drying Shrinkage.—NaOH seemed to increase the shrinkage of bodies containing Mississippi clay and to first decrease and then increase the shrinkage when Dorset clay was used (see Fig. 4). The acid again acted oppositely to that of the alkali.

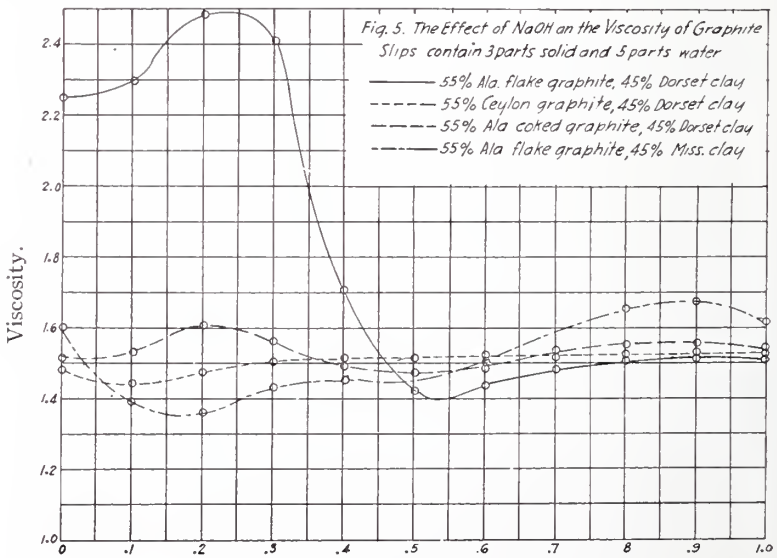
Six No. 60 graphite crucibles, containing one per cent NaOH, in terms of clay, were made. In drying these were set upright, the rims being unprotected. This drying treatment was more severe than that used in practice—where the rims are protected by setting the crucibles upside down during drying. The crucibles showed no signs of cracking—as was noted by Staley[1] in clays containing alkalies. Staley, however, worked with low-grade clays which had a tendency to crack in drying, and, furthermore, the test pieces he used contained 100 per cent clay. In the pres-

[1] H. F. Staley, "The Effect of the Addition of Salts on the Drying Properties of Clays," *Trans. Am. Ceram. Soc.*, **17**, 697–711 (1915). Discussion, *J. Am. Ceram. Soc.*, **1**, 214 (1918).

ent case high-grade clays were used and the bodies contained 60 per cent of non-plastic material—which may account for the difference.



Viscosity.—When NaOH was added to Body No. 5 (containing Dorset clay and Alabama flake graphite) the viscosity was at first increased and when more than 0.3 per cent was added it was decreased (see Fig. 5).



When Ceylon graphite or Alabama coked graphite were used in the place of flake graphite, this increase in viscosity with small additions of electrolyte was not nearly so pronounced. It appears that the effect of flake graphite itself or of some constituents of the graphite (possibly the oils used in flotation) on the clay were factors which caused this increase in viscosity of the slip.

Where Alabama graphite was used with Mississippi clay, the viscosity of the slip was decreased upon adding small percentages of NaOH, this having been most pronounced when 0.2 per cent (in terms of the clay) was used. With this clay no increase in viscosity, such as was obtained with the Dorset clay, was observed.

In attempting to cast body No. 1 with 0.2 per cent electrolyte, it was found that the shrinkage was excessive—causing many cracks in the trial crucibles. Bodies containing 0.3 to 0.4 per cent NaOH gave the best results. When more than 0.5 per cent NaOH was used, the graphite would not remain in suspension and the casting proceeded very slowly. With this content of electrolyte only thin-walled vessels could be cast.

It was noted that bodies containing Ceylon graphite could be casted more easily than those containing Alabama flake graphite.

Summary.

The addition of NaOH to graphite-crucible bodies containing Dorset bond clay spoiled the working properties of the bodies, while the effect of the same electrolyte on bodies containing Mississippi bond clay was to improve their moldability. HCl had an effect opposite to that of NaOH and improved the working properties of the body containing Dorset clay.

The effect of small additions of NaOH to bodies containing Dorset clay was to decrease the dry transverse strength but, where larger percentages of electrolyte were used, the opposite was true. The effect of NaOH on the body containing Mississippi clay was to increase the strength.

An improvement in dry strength was gained by first grinding the flake graphite with the NaOH and water before mixing with the clay.

Small additions of NaOH decreased the density of bodies containing Dorset clay and increased the density of bodies containing Mississippi clay. Where larger additions of this electrolyte were used, the opposite effects were produced. HCl produced opposite effects to that of NaOH.

The effect of small amounts of HCl was to make the body containing Dorset clay more open while adding a larger percentage made the body less porous. NaOH made this same body less porous. The effect of NaOH on bodies containing Mississippi clay was to decrease the porosity when small percentages were used and increase the same when larger amounts were used.

NaOH seemed to increase the shrinkage of bodies containing Mississippi clay and to first decrease and then increase the shrinkage when Dorset clay was used. The acid again acted oppositely to that of the alkali.

When NaOH was added to slips containing Alabama flake graphite and Dorset clay, the viscosity was increased, but upon adding more alkali, it was again decreased. When Ceylon graphite or Alabama coked graphite was used in the place of Alabama flake graphite, this increase in viscosity was not noted. Where Mississippi clay was used in the place of Dorset clay, the effect of small percentages of NaOH was to decrease the viscosity.

Bureau of Mines,
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RADIOACTIVE BRICKS.

By H. J. Knollman.

The manufacture of radioactive clay bricks or briquettes has its chief interest from a ceramic viewpoint in the fact that a clay product is being made and used for an unusual purpose. This, therefore, also entails some work of an unusual nature. So while some of the subject matter of this paper is outside the silicate field, nevertheless the main features fall within the scope of ceramics.

Radium, because of its value, scarcity and wonderful properties, occupies a place of much interest in popular as well as scientific circles. Its market value is about $100 a milligram, or over two and a half million dollars an ounce. There is considerably less than a pound of prepared radium in the world, and in the United States, there is prepared from a few grains up to about 50 grams per year. To produce a gram of radium element, from 400 to 500 tons of carnotite ore are required on an average. It is prepared in this country, chiefly from the carnotite ores of Utah and Colorado. This ore is treated a few hundred times with acids, requiring months of time. A few hundred additional fractional crystallizations must be made before the radium element is obtained in a concentrated form. It may finally be precipitated in a finely divided state as the insoluble radium sulphate, intimately mixed with barium sulphate, from which it is extremely hard to separate. It is about the scarcest article in the world and the most valuable, since not only are the ores scarce, but the yield of the element is so small.

Becquerel discovered the radioactivity of uranium in 1896, and M. and Mme. Curie discovered radium and polonium shortly after. We now have over thirty of these wonderful substances with their almost life-like properties. Of these radium is the most important, followed by uranium, thorium and actinium.

Radioactivity is the spontaneous successive disintegration of radioactive atoms, with emission of "rays." In its characteristic

property of radioactivity, radium is absolutely independent of its environment, whether it be in its natural mineral state, in an isolated state, or mixed with other substances. In all its various compounds it is constantly giving off alpha rays and emanation at a uniform rate, which is not affected by extremes of heat or cold or of pressure, by the strongest reagents, by the action of powerful explosives or intense electrical discharges. The rays emitted from radium are of three types, the alpha, beta and gamma. The alpha rays are positively charged helium atoms shot out in all directions in the form of continuous volleys of tiny projectiles, with velocities of about 12,000 miles per second. They are completely absorbed by a very thin screen or membrane. The beta rays are negative electrons emitted with velocities from one-third to nearly the velocity of light, 186,000 miles per second. They are harder to absorb than the alpha rays, the harder beta rays requiring over a millimeter of lead for their absorption. The gamma rays are not considered to be particles of matter at all, but rather waves in the ether resembling the X-rays. They are the most penetrating form of radiation, the hard gamma rays from radium C requiring over 13 centimeters of lead for their absorption. Over ninety-five per cent of the energy of radiation from radium and its products is in the form of corpuscular alpha radiation, however.

The first disintegration product from radium is emanation, which it constantly gives off. It is an inert gas, similar in its properties to helium and argon, and it obeys all the laws of a true gas. It can be collected, confined and handled in ordinary containers and is quite soluble in water. Emanation in turn disintegrates into alpha particles and radium A, from which in the same way come radium B, radium C, radium D, radium E and radium F, in succession, with varying periods of average life. It is from these products, rather than from radium itself, that the beta and gamma rays are given off.

When a radioactive element changes, it does not as a rule do so once only, but there are several successive changes following one another. Whereas the first change is slow, the subsequent changes often are relatively more rapid. At any stage, the total amount of radioactivity is constant and unaffected, although it

may be divided into several parts. Hence, radioactivity is due to equilibrium between continuous and opposing changes, decay and regeneration. Thus, while the emanation disintegrates or decays, radium is spontaneously manufacturing a fresh supply. So small a fraction of the whole is changing, however, that the main part of the radium will remain unchanged after hundreds of years. The radioactivity of emanation rapidly decays away so that in four days it is one-half as active, in eight days one-fourth as active, and so on, until it is practically zero at the end of a month. The time required for a radioactive body to lose one-half of its activity is known as the period of average life—which for radium is about 1800 years and for uranium, eight million years.

Radium, because of its long life and because of the number of short-lived products which follow it, emitting the different rays, is the most useful radio-element. One of its largest uses is in radium luminous compounds for use on watch dials, compasses and instrument dials. It also finds extensive use in the treatment of dermatological growths, and in the therapy of malignant growths. Particularly in cancer, it is used externally, the hard beta and gamma rays being utilized for this purpose.

It is for internal use, however, that radium emanation has become important as a therapeutic agent. Water charged with radium emanation, when taken, scatters this emanation through the body and seems to arouse all the cells of the body to a greater activity, being a sort of tonic to them all. In general, the emanation increases the activity of certain enzymes, promotes tissue oxidation and the general metabolism, and helps in the elimination of waste products. It is an effective agent in the treatment of such diseases as gout, arthritis, rheumatism, neuralgias, neuritis, anemia, and troubles of this nature. Many radioactive springs containing mere traces of mineral solids have long enjoyed a reputation in the treatment of various diseases of this kind.

Although various methods have been adopted for the introduction of radium emanation into the body, the drinking of radioactive water, natural or artificial, is the simplest. Natural radioactive springs are charged with radium emanation by contact with radium minerals. These natural waters when bottled and transported lose their activity speedily, owing to the rapid disintegra-

tion of the emanation. The artificial method has the advantage of providing a source for the renewal of the emanation from day to day. By using an insoluble salt of radium, it can be used repeatedly and its use continued indefinitely.

The most satisfactory and simplest method of charging water with emanation is to incorporate definite amounts of the insoluble $RaSO_4$ in a finely subdivided state throughout the clay, mold or press into briquettes or rods, and then burn them. When placed in water in tightly closed bottles, these bricks or plates, as they are generally called, give off the emanation, which dissolves in the water, the quantity depending upon the radium content and the time of submersion. Since radium is a very permanent body, losing only one-half its weight in about eighteen hundred years, these plates retain their activity indefinitely.

The strength of radioactive water is usually given in Mache Units per liter. A Mache Unit equals 0.001 electrostatic unit, one of which equals 3.33×10^{-10} amperes, and is the standard unit adopted. Bricks of different strengths are manufactured, the usual strengths varying from 500 to 5000 Mache Units each per 24-hour yield. These will take a corresponding amount of radium element, varying from a few to several hundred micrograms.

While a given amount of radium always gives off a uniform amount of emanation, the proportion given out by the insoluble salt depends largely upon its state of subdivision. In dense, compact form, radium sulphate will only yield $2^1/_2$ per cent, while if it is finely powdered and spread so that it presents a large surface, 10 per cent can be obtained. This latter result can be obtained in a clay body with a fairly large absorption, since the amount of surface is then at a maximum. Every part of the radium sulphate, not the surface only, is giving out the three types of rays, and the alpha rays especially will be absorbed by the dense and heavy radium sulphate, unless it is spread out. The $RaSO_4$ is, therefore, precipitated along with the $BaSO_4$ in a finely divided state, thoroughly dried, and is then ground to an almost impalpable powder.

The basis of "Radio-Rem" bricks or plates, as they are gen-

erally called, is Albany slip clay—more than 50 per cent of the body being made out of this clay. Besides the Albany clay, a rather sandy non-plastic clay is used and also some infusorial earth. The purpose of the infusorial earth is to cut down excessive plasticity, and also to render the body more porous. To these clays, the radium-barium sulphate is added in varying amounts, depending upon the radioactive strength desired. These clays are each ground dry so as to pass a 40-mesh sieve before being used. If any broken plates are used in the mix, they are ground up to a like fineness. As a matter of fact, the composition of the body is relatively unimportant. Any good clean clay, with a low amount of soluble salts, and maturing with the addition of less plastic clays and non-plastic to a hard body at about cone 08, can be used.

The prepared clays are then weighed out, the total check weighed, the radium-barium sulphate is added, and the whole is placed in ordinary porcelain jar mills with enough flint pebbles to insure thorough mixing. The total weight per batch in the mill seldom exceeds 1000 grams, which is equivalent to 25 plates. The whole is mixed dry for a few hours, is then removed from the mill and the pebbles carefully brushed off. Owing to its value and the fact that a minute amount of the radium element goes a long way in its effects, great care must be exercised throughout the process of manufacture to avoid even very small losses.

The mixture is then check-weighed so that the weight corresponds to the original weight. Water is then added and the mixture thoroughly worked up with a large spatula to a uniform crumbly mass. From 26 to 27 per cent water is required for proper consistency, the best consistency being midway between that required for the dry-press process and the stiff-mud process. This is an important step, as just a little too much or not quite enough water results in an inferior product. If too little water is used, the pressed body will be crumbly, will break and crack easily, and will burn soft and punky at this temperature. If, on the other hand, just a little too much water is used, the mix will ball up into lumps, the plate when pressed will be too soft to handle, and it will also burn soft and punky at cone 08. Any large lumps in the mix are broken up and the entire mass is made as

uniform as possible. If not obtainable otherwise, the mix can be made uniform by passing it through about a 16-mesh sieve.

The total weight is next obtained and the exact percentage of water is then calculated. The basis of the calculation is 40 grams dry weight per plate. Each individual plate is then weighed up to the nearest tenth of a gram, the main batch being kept covered with a tight fitting lid as much as possible. By so doing, very little drying out takes place and the mixture is kept uniform in moisture content until the entire batch is weighed up. The plates are pressed on a hand press, care being taken to spread the mixture as uniformly as possible in the die before pressing. The dimensions of the die are $3^3/_4'' \times 1^1/_4'' \times {}^3/_8''$, with corrugated surfaces on the top and bottom sides. This is also the approximate size of the burned plate, since the shrinkage is comparatively slight. The plates are then dried out, no special precautions being necessary. Owing to their small size, it is seldom necessary to use a dryer and open air drying is sufficient.

The plates are burned in small laboratory kilns of the muffle type, such as the R. & H. China Muffle or Caulkins Muffle kiln. They are burned to cone 08 in from eight to ten hours, and the same length of time is required in the cooling. Natural gas is preferred, although any fuel may be used, provided the burn is kept thoroughly oxidizing. Care must be taken to maintain thorough oxidizing conditions, for if there is any reduction whatever, the $RaSO_4$ is easily reduced to $RaSO_3$, which is soluble in water and the radium salt, together with its emanation, is lost after the first immersion of the plate in water. Flashing must be avoided for the same reason.

Best results are obtained when the plates are burned between cones 010 and 06, cone 08 giving most satisfactory results. Above cone 06, the yield in emanation decreases. At cone 1, only about 30 per cent of the activity is obtained. Since, however, a given amount of radium always gives off a fixed definite amount of emanation and alpha rays, no matter what is its environment, the emanation must be absorbed or occluded in some way. This is what happens at cone 1. The plates must be burned at a low temperature so that a minimum amount of fluxing action takes place—just enough to give a good commercial hard body which

will stand a little rough usage. The presence of glassy constituents, fluxes of iron, etc., forms an impervious seal to the alpha rays, so that they are kept within the plate. The water does not penetrate these sealed pores, and since the alpha rays are stopped by a thin membrane, the effect of the alpha radiation in such instances is lost. Since 95 per cent of the energy of radiation is in the form of the alpha radiation, this effect may be considerable. Reduction in the radioactivity was especially noticeable in some trial whiteware bodies burned to cones 1 and 3, in which a large amount of feldspar and cornwall stone were used.

The plates upon removal from the kiln are washed in running water for several hours in order to eliminate any soluble salts that may be present. They are then dried and are ready for testing. When used by the patient, the plates are placed in a tightly stoppered bottle of about one-half pint capacity, the bottle is then completely filled with water and the stopper inserted. The water is then drunk by the patient at specified intervals.

It was thought for some time that the absorption and porosity of the plates played a very important part in the yield of emanation, as expressed in Mache units. More exhaustive researches showed, however, that the standardization of the radium-barium salt used, the degree of fineness of the salt and materials, the temperature of burning, and the final testing of the burned plate had a much more important effect than the porosity of the plate. All other factors being constant, the yield is the same, no matter what the porosity, provided the plate is porous enough to allow the water to penetrate thoroughly through the entire plate. At cone 08, absorptions ranging from 15 to 40 per cent gave the same yield in Mache units per day, provided all the materials used were the same, and the range in composition was not great enough to materially affect the vitrification.

One of the chief effects of radioactive bodies and their disintegration products is to produce an ionization of the atoms of whatever substance the rays penetrate. Hence, any electrified object has its electricity rapidly discharged in the neighborhood of a radioactive substance. The instrument generally employed to detect this effect is the gold-leaf electroscope, which is capable of so great refinement that it affords the most delicate and sensitive

test that it is possible to employ for the detection of radioactivity. This principle is made use of not only in testing the strength of the radium salts used, but also to test the burned plates, or rather the radioactivity of the water which has been in contact with the plates. The radioactivity of the water is measured by a fontactoscope, which is an electroscope with a chamber for ionized air and a scale for measuring and timing the discharge. The fontactoscope is first standardized by testing a solution of a known amount of radium element, which has been sealed for thirty days, as it is then in radioactive equilibrium. After a standard has been obtained, the radioactive strength of any water containing emanation can be tested, measured and calculated.

Great care must be exercised in sampling the water. When water with emanation in solution is shaken up or otherwise disturbed, from 10 to 30 per cent of the emanation in the water may be lost, due to diffusion of the emanation from out of the water. When tested, the water is first carefully drawn from the bottle by means of a vacuum into a closed container, where it is then purposely shaken up to release the emanation from the water, from where it is then pumped into the ionization chamber of the fontactoscope. Then at definite specified time intervals, corresponding to the time intervals of the standard, the gold leaf is given a static charge produced by the simple ordinary method of rubbing two dissimilar bodies together and separating them. As the gold leaf collapses, due to the ionization produced by the radium rays, it moves across the scale in the fontactoscope. The time required for it to move across this scale is the time of discharge, which is obtained by the use of a stop-watch. The strength in Mache units per day is then calculated.

Special precautions in the testing of the plates must be taken or else erratic if not erroneous results will be obtained. No tests should be made upon an immersed plate until the third or fourth day, the results being too low on the first few days, since radioactive equilibrium has not been established, the period of average life for emanation being 3.8 days. The bottle containing the plate to be tested must be emptied and refilled with fresh water at every 24-hour interval. If refilled in a shorter time interval, the water remaining in the pores of the plate, which is not in-

considerable, will not have a full 24-hour emanation charge, so that if a test is made on the succeeding day, low results will be obtained, due to this factor. Likewise, if a plate is left in the water longer than 24 hours, and the bottle is then refilled, it cannot be tested accurately on the succeeding days, since the pore water contains more than a 24-hour charge, causing the resultant readings to be too high. These troubles are avoided by refilling the bottle exactly every 24 hours.

The method of charging the gold leaf in the fontactoscope is important. It should always be charged just sufficiently to bring it over to the extreme edge of the scale. It should be brought over in one charge, and not be charged in steps, so as to swing the gold leaf back and forth across the scale, for in so doing, the rate of ionization is retarded or interfered with, causing the speed of collapse of the leaf, or rather the rate of discharge to be slow, thus giving a low result. High humidity has the same general effect. When the humidity is high, the charging of the gold leaf becomes so difficult that if it is charged at all it will discharge erratically, giving unreliable results.

Although the manufacture of radioactive bricks presents no special difficulties from the ceramic standpoint, the outstanding factors are the accuracy and extreme care required to avoid even small losses in each step of the process of manufacture, as well as the precautions necessary for the correct testing of the bricks, all of which have a direct bearing upon the amount of radium element necessary to produce a plate of definite strength in Mache units per day.

SCHIEFFELIN & CO.,
NEW YORK, N. Y.

COMMUNICATED DISCUSSIONS.

CHAS. H. VIOL: The results obtained by the London Radium Institute would seem to indicate that the use of very strong radium emanation solutions is advantageous, their dosage for chronic arthritis being the daily administration of one-fourth liter of radium emanation water containing not less than one millicurie of radium emanation per liter, this being equivalent to about 700,000 Mache units of radium emanation per dose per day.

Our own inclination has been to recommend a dosage inter-

mediate between this high dosage of the London Radium Institute and the low dosage which can be obtained by the use of radioactive bricks; and to prepare such emanation solution most economically we have preferred to place the radium salt in a solution, securing the emanation in aqueous solution by a suitable arrangement of vessels which permits of the radium emanation being passed in a closed circuit, bubbling through the water which is to be made radioactive. This method permits of the utilization of fifty per cent or more of the emanation produced, and is consequently at least four times more effective in this respect than the radioactive bricks.

STANDARD CHEMICAL CO.,
PITTSBURGH, PA.

H. J. KNOLLMAN: While it is true that the method of obtaining emanation from a solution of a radium salt and bubbling it through the water to be drunk is more economical, yet it is not at all a practical method for the ordinary person who wants to have radioactive water at home or who wishes to carry the apparatus around with him. The method of immersing radioactive bricks in tightly stoppered bottles provides a convenient permanent source of emanation, which can be obtained by the patient at any time or place. It is also quite possible to produce radioactive bricks of considerably higher strengths than those given as being the usual strengths of bricks manufactured.

SOME PROPERTIES OF BOND CLAYS FOR GRAPHITE CRUCIBLES.[1]

By M. C. Booze.

Introduction.

During the war, the problem of securing materials suitable for the manufacture of graphite crucibles became rather acute, due both to the reduction in importation of foreign graphite and to the inability of crucible manufacturers to obtain certain clays which they had used and considered necessary.

In connection with work being done by the Bureau of Mines upon graphite crucibles, it was decided that more complete data than was available upon bond clays for use in graphite crucibles was necessary. Although considerable work has been done upon bond clays[2] and plastic fire clays with respect to their burning properties and their bonding strength when used with sand or clay-grog, practically no results are available from investigations in which graphite was the non-plastic material used. Bleininger's work upon bond clays is very valuable to the graphite crucible manufacturer in showing the temperature-structure relations of the clays.

The characteristic properties of graphite are decidedly different from those of sand or calcined grog. The graphite is infusible at clay-burning temperatures, although it burns readily in air at temperatures near or above 900° C. It thus precludes a reducing atmosphere in contact with the bonding clay.

The graphite may also have a mechanical effect upon the strength of the resultant body, unlike that produced by the more nearly spherical particles of fire-clay grog, in that the flakes always lie with the planes of their broad faces perpendicular to the line of pressure applied during molding. The graphite grains also have well defined cleavage planes along which they

[1] By permission of the Director, U. S. Bureau of Mines.

[2] Bleininger and Loomis, "The Properties of Some American Bond Clays," *Trans. Am. Ceram. Soc.*, **19**, 601 (1917).

are readily divided. The surfaces of the grains are not easily wet by water and the particles have somewhat the quality of oiled plates. These characteristics are not inherent in other non-plastics, such as sand or fire-clay grog, and would appear to have an injurious effect upon the strength of the finished body—due to the formation of laminations and to the poor adherence of the clay to the graphite surfaces. It is also noticeable that the clays when mixed with graphite are not as readily affected by heat as are the clays alone.

Since the graphite is quite unlike other non-plastics, it was thought advisable to determine some of the more important properties of bond clays when used with graphite alone. The clays for graphite crucibles may be classified, according to their use, into two classes: first, those suitable for brass-melting crucibles, and second, those suitable for steel-melting crucibles. Clays of the first class must burn fairly dense and develop good strength at or near 1100° C and should retain the same structure at the highest temperature used for brass melting, about 1250° C.[1] Clays of the second class must necessarily be more refractory than those of the first class—since crucibles for steel melting are used at temperatures ranging from 1400° to 1600° C. They must possess in this temperature range about the same qualities as those of the first class do between 1100° and 1250° C.

The crucible body must be dense enough at the pouring temperature of the metal to prevent penetration of slags and molten metal and to prevent oxidation of the graphite. However, to partly nullify the effect of strains set up by sudden heating and cooling of the crucible at every heat, the body should not be exceedingly dense. The graphite, however, tends to equalize the cooling strains and Seger[2] states that feldspar acts in the same direction. The high per cent of non-plastics used also decreases the effect of abrupt heat changes. Feldspar is also used in crucibles for brass melting to reduce the temperature at which the bodies become dense. Seger[3] found that Klingenberg clay,

[1] H. W. Gillet, "Brass Furnace Practices," Bureau of Mines, *Bulletin* **73**, 129.

[2] H. Seger, "Collected Writings," **2**, 1136.

[3] *Ibid.*

which has been largely used for crucibles, contained, in the crude state, about 6 per cent of feldspar. This is also confirmed by an analysis given by Bleininger.[1] The refractoriness of bodies used in crucibles for steel melting is often enhanced by the addition of plastic kaolin or sand, or both.

Investigation.

The clays tested were ground to pass a 20-mesh screen and mixed with an equivalent amount by weight of No. 1 grade Alabama graphite which had the following screen analysis through Tyler standard testing sieves:

	Per cent.
On 20-mesh	0.05
Through 20-mesh, on 35-mesh	7.55
Through 35-mesh, on 48-mesh	24.50
Through 48-mesh, on 65-mesh	48.72
Through 65-mesh, on 100-mesh	17.99
Through 100-mesh	1.14

The Alabama graphite had a specific gravity of 2.29 and an ash-content of 5.26 per cent.

Nineteen domestic, one English, and one German clay were tested. Some domestic clays were not included because they could not be obtained; some arrived too late; others were not tested because we were unable to ascertain the localities from whence they came. A few blended clays were offered for tests but as we were unable to learn the identity of the individual clays which made up the blends, the tests would not have had much value and were not made.

The tests made covered water of plasticity, resistance to slaking, ratio of pore to shrinkage water, ratio of pore water to true volume of the body constituents, a classification of the clays as to working qualities when used with graphite, dry and burned transverse strength, the degree in which the clay aids in resistance to oxidation of the graphite, loss in strength from sudden cooling, and the softening points of the clays alone.

[1] "Notes on the Crucible Situation," read before the Institute of Metals, Boston, Mass., Sep 25, 1917.

Clays Used.—The following were the clays tested, the numbers assigned to them in the investigation, and the dealers from whom they were obtained:

No. 3.01 From Laclede-Christy Clay Products Co., St. Louis, Mo.
No. 3.02 From Laclede-Christy Clay Products Co., St. Louis, Mo.
No. 3.03 From J. T. Bramlett, Enid, Miss.
No. 3.04 From J. T. Bramlett, Enid, Miss. (from property of Southern Ball Clay Co.)
No. 3.05 From McClendon Clay Co., Senatobis, Miss.
No. 3.06 From F. E. Bausch, St. Louis, Mo.
No. 3.07 From F. E. Bausch, St. Louis, Mo.
No. 3.08 From F. E. Bausch, St. Louis, Mo.
No. 3.09 From F. E. Bausch, St. Louis, Mo.
No. 3.10 From Harbison-Walker Refractories Co., Pittsburgh, Pa.
No. 3.11 From Harbison-Walker Refractories Co., Pittsburgh, Pa.
No. 3.12 From Harbison-Walker Refractories Co., Pittsburgh, Pa.
No. 3.13 From Highlands Fire Clay Co., St. Louis, Mo.
No. 3.14 From J. T. Bramlett, Enid, Miss.
No. 3.15 From Mandle Clay Mining Co., Porters, Tenn.
No. 3.16 From Kentucky Cons. & Improvement Co., Mayfield, Ky.
No. 3.17 From Kentucky Cons. & Improvement Co., Mayfield, Ky.
No. 3.18 From Kentucky Cons. & Improvement Co., Mayfield, Ky.
No. 3 19 From Mandle Clay Mining Co., Whitlock, Tenn.
No. 3.20 Dorset English ball clay from John Sant & Sons Co., E. Liverpool, Ohio.
No. 3.22 Klingenberg crucible clay from Jos. Dixon Crucible Co., Jersey City, N. J.

Preparation.—The clays were mixed dry with the graphite, water added to the best working consistency as determined by feel, pugged thoroughly by hand, wrapped in a damp cloth and laid away in a covered jar for two days, after which they were taken out, re-pugged and molded.

Two one-inch cubes for the slaking test were made from each mix together with a sufficient number of bars, $1/2'' \times 1''$ in cross section, for transverse strength determinations. Cylindrical oxidation trials $4''$ long by $2''$ in diameter with semi-spherical ends were also made. In molding the pieces, bars somewhat narrower and deeper than the mold were cut from the plastic mass with a wire-bow, placed in the mold and pressed down by hand, smoothed off with a spatula, and numbered.

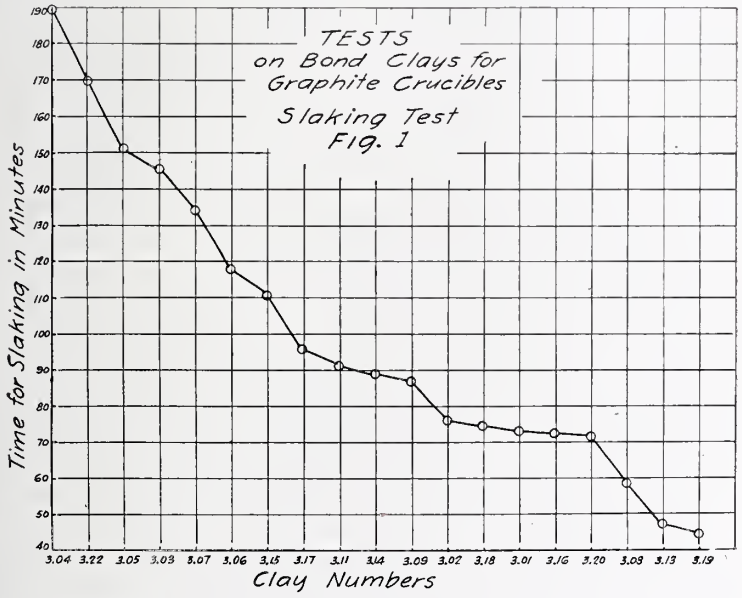
All of the pieces were dried for two days under a cloth, the cloth

being then removed and the bars dried for several days at room temperature, then for two days at 70° C, and finally for 24 hours or longer at 110° C.

The wet weights and wet volumes of three bars were determined immediately after molding. The dry weights and dry volumes of the same three bars were determined after the final drying at 110° C. The water of plasticity was expressed in the per cent loss in weight in terms of the dry weight. As a rule, the more plastic clays required a higher proportion of tempering water than did the lean ones.

The wet and dry volumes were determined in a volumeter of the pycnometer type as described by Schurecht.[1] The drying shrinkage was expressed as the per cent loss in volume in terms of the dry volume of the bar.

The rate of slaking was taken as the time, in minutes, for disintegration in water of a one-inch cube previously dried at 110° C. The result taken in each case was the average of two determinations. (See Table 2 and Fig. 1.)



[1] *J. Am. Ceram. Soc.*, 1, 556 (1918).

The very plastic clays dried to dense horn-like bodies and required longer periods for disintegration than did the lean clays which gave more porous and friable bodies. The test, however, is subject to considerable error on account of the large number of factors which cannot be closely guarded. It serves only in differentiating between clays of widely divergent bonding properties.

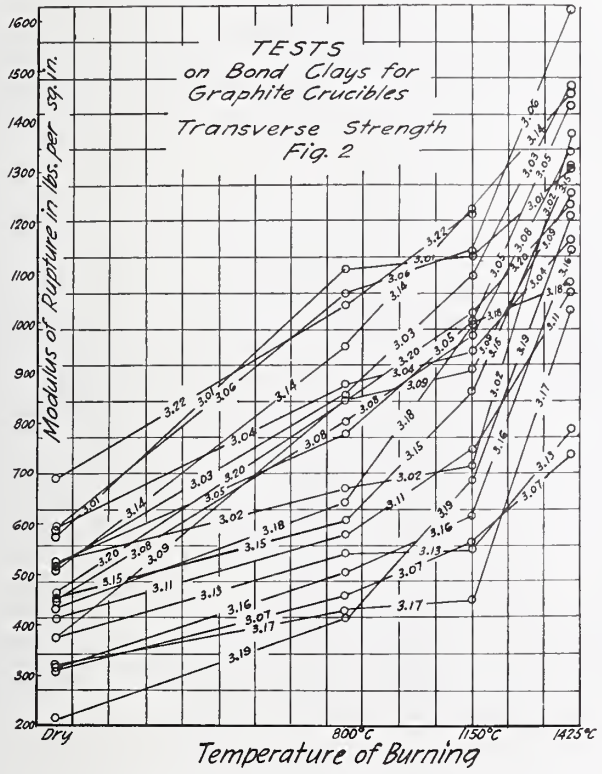
The dry transverse strength was taken as the modulus of rupture in pounds per square inch. Bars $^1/_2'' \times 1''$ in cross section were broken on a five-inch span by a uniformly increased load applied at a point equidistant from the bar supports. The dimensions at the break were measured to the nearest $^1/_{100}''$ by means of an Ames dial. The moduli were then calculated from the modulus of rupture formula. The results given (Fig. 2) are the average of fifteen determinations in each case.

Burning.—Bars for burned transverse strength, bars for the determination of loss in strength from sudden cooling, and the oxidation trials were embedded in graphite in covered saggers and burned in horizontal draft gas-fired test kilns. A sufficient number of bars of each mixture to cover all requirements were fired in individual burns to each of the following temperatures at the rate of 40° per hour: 800°, 1150°, and 1425° C. These temperatures correspond, respectively, to the temperatures at which the crucibles are fired, and to the temperatures at which they are used in brass and steel-melting. Upon reaching the temperature desired, the kiln was held for a period of four hours.

The transverse strength for each temperature at which the bars were burned was determined by breaking on a three-inch span and is expressed in pounds per square inch (Fig. 2).

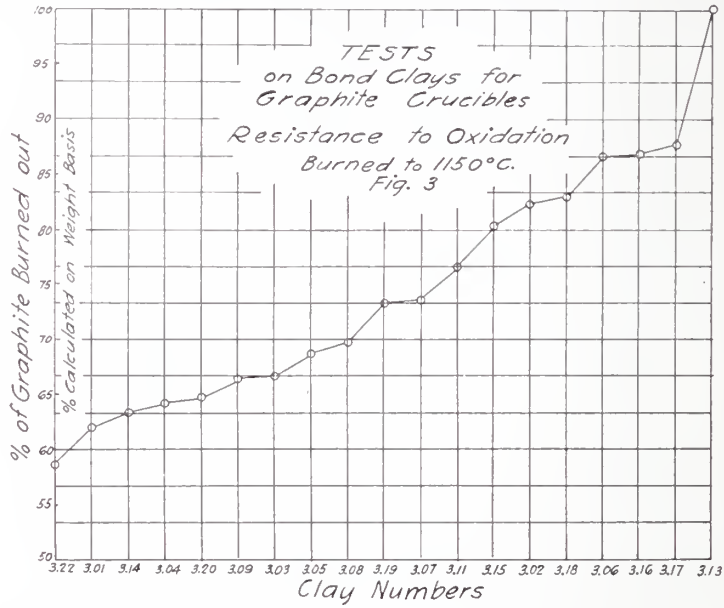
The four oxidation trials from each mixture—two having been burned to 1150° and two to 1425° C—were weighed and placed about one-inch apart in a gas-fired test kiln. The pieces were so supported on knife edges that they could be completely encircled by the furnace gases. The temperature was increased to 1100° C in $13^1/_2$ hours and held for two hours. The content of carbon dioxide in the flue gases was maintained at 7 per cent throughout the burn by means of a Simmance-Abady CO_2 recorder. Upon cooling, each piece was again weighed, sawed at right angles to

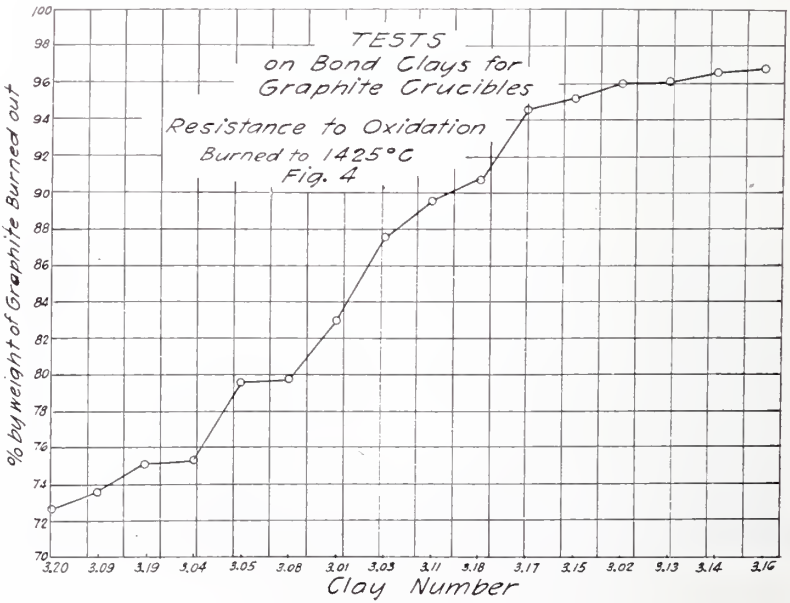
the longer axis, and both the unoxidized area and the area of the whole piece measured with a planimeter. The oxidized area was expressed in per cent of the whole area.



In order to determine the true loss in weight in per cent of graphite present, there must be taken into account both the ash-content of the graphite and the weight lost by the clay in dehydrating—since the clay at the time the pieces were molded was in the hydrated condition and the loss of the chemical water left a larger proportion by weight of the graphite than was originally present. The loss from dehydration was assumed at two molecules of water for every molecule of clay as first used, or 13.9 per cent of the original weight of the clay. The ash-content of the graphite used was 5.26 per cent. From the data, it was

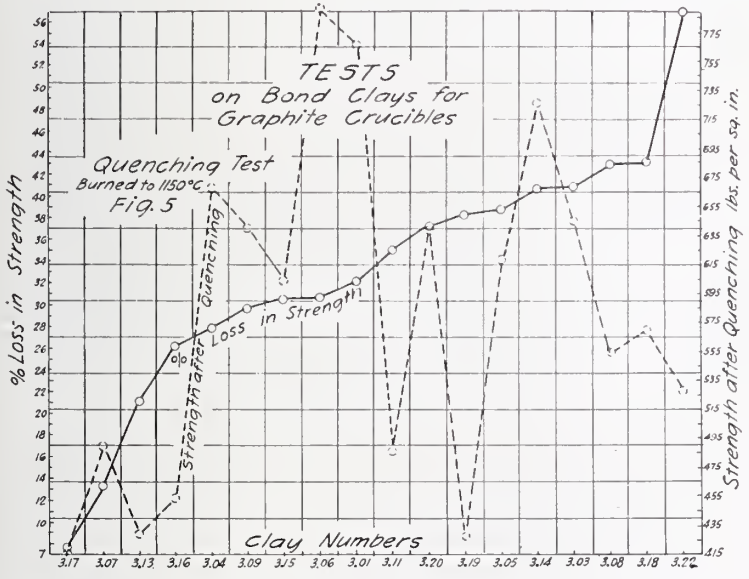
then possible to calculate the per cent of the graphite oxidized in terms of the graphite originally present (Figs. 3 and 4).





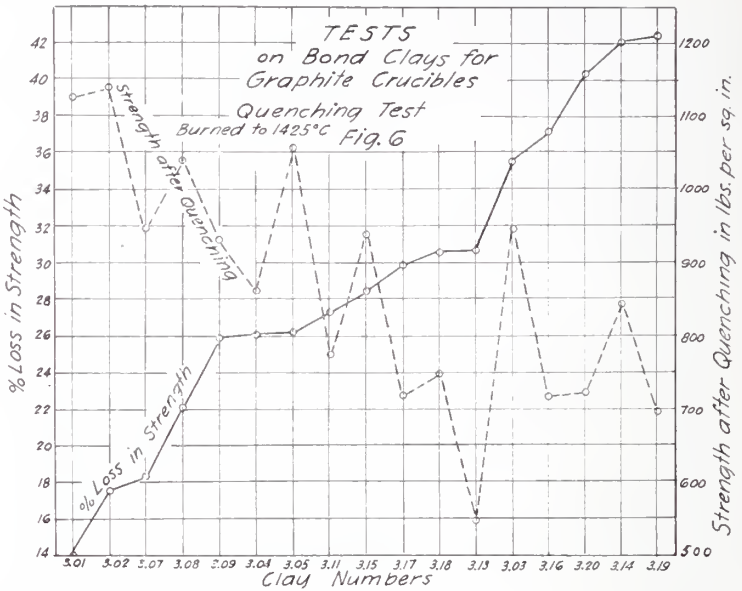
Oxidation trials such as were used, with no sharp corners, insure an even penetration of the oxidizing gases over the surfaces and give more reliable data than would be the case if rectangular pieces were used.

The loss in strength from sudden cooling was determined upon bars previously burned to 1150° and 1425° C, by heating to 800° C in an electric furnace and plunging into water at a temperature of 18° C. The bars were then dried and the operation



repeated until they had been quenched five times. The moduli of rupture of the bars were then determined by breaking on a three-inch span as before and the per cent loss in strength in terms of the original strength was calculated from the available data. The results (Figs. 5 and 6) are the averages of at least 12 determinations in each case.

The softening points of cones made from the individual clays were compared to those of standard cones in a gas-fired pot furnace, and were expressed in terms of the latter cones (Table 3).



The ratio of pore to shrinkage water was calculated in each case from the data at hand (Table 2). The value of the ratio was obtained by means of the expression

$$\frac{W - D}{Vw - Vd} - 1$$

where

W = wet weight of the piece.
D = dry weight of the piece.
Vw = wet volume of the piece.
Vd = dry volume of the piece.

The ratio of pore volume to the true volume of the bar constituents is more important and the volume of the ratio may be obtained by the expression

$$\frac{(W - D) - (Vw - Vd)}{\frac{D}{S}}$$

where

W = wet weight of the bar.
D = dry weight of the bar.

Vw = wet volume of the bar.
Vd = dry volume of the bar.
S = true specific gravity of the bar.

The speeific gravity of eaeh elay was taken as 2.6 and the specifie gravity of the graphite used was found to be 2.29, so that S in the above expression has a value of 2.445.

To determine the order in whieh the clays might be elassed as regards working qualities, 500 grams of a 1 : 1 elay and graphite mixture was made up from eaeh elay, tempered and pugged by the same man in eaeh ease, and aged and elassified by four men individually. The elays were then elassed as seemed most eonsistent with the results. (See Tables 1, 2, 3 and 4.)

TABLE 1.

Clays arranged in the order of their working qualities.[1]

3.08	Warps considerably in drying.	Very good working qualities.
3.03	Warps considerably in drying.	
3.22	Warps considerably in drying.	
3.20	Warps considerably in drying.	
3.06	Bars dry straight, but have peculiar wrinkled surfaces.	
3.04	Warps considerably in drying.	
3.09	Warps considerably in drying.	Good working qualities.
3.19	Dries fairly straight.	
3.14	Warps somewhat.	
3.01	Warps somewhat.	
3.05	Warps somewhat.	
3.18	Dries well.	
3.15	Warps only a small amount.	
3.16	Dries well.	Rather poor working qualities.
3.07	Dries well.	
3.11	Warps very little.	
3.02	Warps very little.	
3.13	Dries well.	
3.17	Warps very little.	
3.10	Dries well.	Too lean for molding.
3.12	Dries well.	

[1] No trouble from cracking was experienced with any of the clays.

TABLE 2.

No.	Water of plasticity.	Drying shrinkage in terms dry-bar volume.	Ratio, pore to shrinkage water.	Ratio, pore to true volume of the bar.	Time for slaking, in minutes.
3.01	28.31	22.59	1.117	36.5	73
3.02	36.85	22.72	1.709	56.2	76
3.03	29.34	21.32	1.24	39.8	146
3.04	27.75	22.31	1.120	35.9	190
3.05	27.73	21.48	1.168	36.5	151 1/2
3.06	29.48	20.66	1.318	41.1	118
3.07	27.88	15.89	1.885	45.5	134 1/2
3.08	33.42	29.68	0.845	37.4	58 1/2
3.09	34.48	25.57	1.208	46.1	87
3.11	28.65	30.12	1.198	46.65	91 1/2
3.13	25.56	23.91	0.867	28.9	47 1/2
3.14	26.17	18.98	1.360	36.9	89
3.15	35.97	24.71	1.298	49.9	111
3.16	28.56	15.39	1.955	46.4	72 1/2
3.17	29.18	19.71	1.383	41.35	96
3.18	34.08	24.10	1.200	45.4	74 1/2
3.19	27.95	18.64	1.438	40.35	44 1/2
3.20	31.17	23.38	1.178	40.64	71 1/2
3.22	32.02	29.54	0.879	39.16	170

Discussion of Results.

It is evident that no clay satisfies, to a high degree, all requirements for graphite crucible use. In fact, some of the necessary qualities are so diametrically opposite as to insure no one clay being entirely satisfactory. Some of the clays of course rank higher than others as regards all requisite properties and by choosing one of these as the basis and blending others with it, a mixture may be built up which should give more satisfactory results than could be obtained by the use of any single clay.

By a comparison of the data herein given with that given by Bleininger,[1] upon the strength of 1:1 mixtures of bond clay and sand, it may be seen that the average strength of bodies in which graphite is used is considerably higher than that of those in which sand is used. This is probably due to the difference in

[1] *Trans. Am.Ceram. Soc.*, **19**, 601 (1917).

TABLE 3.

No.	Dry strength.	Strength after burning at 800° C.	Strength after burning at 1150° C.	Strength after burning at 1425° C.	Strength after burning at 1150° C and quenching 5 times.	Strength after burning at 1425° C and quenching 5 times.	Per cent loss in strength after burning at 1150° C and quenching.	Per cent loss in strength after burning at 1425° C and quenching.	Softening point in standard cones.
3.01	576.4	1104.11	1131.9	1308.8	768.31	1125.1	32.1	14.0	31
3.02	525.3	671.55	719.2	1378.8	...	1137.0	...	17.5	23
3.03	516.6	857.2	1092.0	1470.4	646.2	948.6	40.85	35.6	31
3.04	590.6	878.1	946.5	1165.5	682.6	861.9	27.9	26.1	30 1/4
3.05	518.3	781.4	1008.9	1433.6	617.8	1058.7	38.8	26.2	30
3.06	595.8	1058.8	1142.4	1618.8	793.1	....	30.6	....	30 1/2
3.07	312.1	460.21	564.6	734.0	489.1	599.5	13.35	18.35	32 1/2
3.08	449.2	804.1	972.1	1339.8	554.8	1043.4	42.9	22.1	30 1/4
3.09	378.0	848.45	910.9	1259.1	640.9	933.9	29.62	25.9	32 1/2
3.11	416.2	580.5	750.7	1065.7	486.8	775.2	35.1	27.3	31
3.13	372.1	542.4	549.2	789.9	429.7	547.0	21.8	30.7	31
3.14	510.9	956.3	1226.1	1459.5	727.7	845.7	40.65	42.1	30 1/2
3.15	453.8	611.3	868.9	1315.3	603.7	940.5	30.5	28.5	32 1/4
3.16	313.8	506.6	619.8	1143.5	457.6	718.0	26.2	37.2	30 1/4
3.17	314.7	432.1	451.1	1026.9	416.5	719.3	7.7	29.9	30
3.18	431.9	644.0	1000.7	1080.0	569.9	749.6	43.1	30.6	33
3.19	214.1	414.8	687.1	1212.0	423.4	698.2	38.3	42.4	32 1/2
3.20	464.5	846.0	1020.0	1234.5	640.1	724.8	37.2	41.3	33
3.22	691.7	1035.1	1219.5	....	526.4	....	56.8	...	31 1/2

TABLE 4.

Oxidation trials burned at 1150° C. Oxidized for 10 hrs. at 1100° C.				Oxidation trials burned at 1425° C. Oxidized for 10 hrs. at 1100° C.		
No.	Per cent of whole area oxidized.	Per cent loss in weight.	Per cent graphite burned out.	Per cent of whole area oxidized.	Per cent loss in weight.	Per cent of graphite burned out.
3.01	94.40	31.53	62.0	70.14	42.2	83.0
3.02	71.59	41.97	82.4	95.01	48.53	96.0
3.03	46.53	33.29	66.6	56.03	44.74	87.6
3.04	47.59	32.76	64.25	62.19	38.37	75.35
3.05	52.65	34.65	68.1	74.76	40.54	79.60
3.06	56.12	43.88	86.6	broken	broken	
3.07	62.93	37.26	73.60	broken	broken	
3.08	76.15	35.29	69.9	67.96	40.55	79.75
3.09	49.60	33.81	66.4	58.34	37.42	73.60
3.11	60.71	39.07	76.62	81.57	45.58	89.6
3.13	96.13	50.91	100.01	97.86	48.88	96.1
3.14	51.67	32.50	63.3	71.87	49.14	96.6
3.15	67.85	41.01	80.4	86.76	48.58	95.2
3.16	87.46	44.15	86.8	96.49	49.48	96.8
3.17	78.32	44.66	87.7	93.99	48.17	94.6
3.18	70.53	42.23	83.0	75.76	46.21	90.75
3.19	54.75	37.36	73.35	65.50	38.31	75.1
3.20	53.43	32.93	64.7	50.0	36.96	72.6
3.22	40.94	29.86	58.7	...	...	...

the shape of the grains. Similar results were obtained in laboratory tests to determine the effect of grain-size and shape upon the dry and burned strength of clay-graphite mixtures. It was found that the angular grains of Ceylon graphite, and the thick grains produced by coking Alabama graphite, gave bodies of lower strength than did the thin flakes of the untreated Alabama graphite.

An attempt was made to determine the strength, at furnace temperatures, of bars made from the clay-graphite mixtures but the bars were oxidized to such an extent, even under as reducing an atmosphere as could be maintained, that this part of the investigation had to be postponed pending the discovery of a suitable protective coating for use on the trial pieces to prevent oxidation.

Accurate data upon the hot strength of crucible mixtures would be very valuable—since the crucibles while at furnace temperatures are subjected to a high internal pressure from the molten metal.

The porosity-temperature and volume shrinkage-temperature relations of the mixtures were also determined but such small changes occurred over comparatively large temperature intervals, that sufficiently accurate data was not obtained from the number of trials employed. This part of the investigation is now being repeated. The data obtained, however, indicates that the refractoriness of the clay is increased by the addition of graphite.

The porosity-temperature relation is very peculiar. In the preliminary laboratory work it was found that the porosities of clay-graphite bodies burned to cone 15 were greater by about 3 per cent of the dry volume than were the dry porosities. From the oxidation curves it may also be seen that the bodies burned to 1425° C were less resistant to oxidation of the graphite than were those burned at 1150° C. This would indicate greater porosity at the higher temperature than at 1150° C. Such an increase in porosity is contrary to the general behavior of clays. even at their over-burning temperatures, as may be seen by consulting the data given by Fulton and Montgomery[1] or by Bleininger.[2]

By a consultation of the data given in Table 3, it may be seen that several of the mixtures after being burned at 1425° C showed a lower proportional decrease in strength from quenching than they did when burned at 1150° C. This may also be due in part to the greater porosity of the bodies burned to the higher temperature.

The peculiar influence of the graphite upon the body may be due to changes occurring within the graphite itself.

It is possible that, since graphites were probably formed under conditions of extreme heat and pressure, an increase in volume occurs upon being heated to a high temperature at atmospheric pressure The volume increase would tend to disrupt the grains

[1] *Trans. Am. Ceram. Soc.*, **17**, 409 (1915).

[2] *Loc. cit.*

along their cleavage planes and so open up the body to some extent.

Mining Experiment Station,
Bureau of Mines,
Columbus, Ohio.

COMMUNICATED DISCUSSION.

M. G. Babcock: The results which we have obtained at this laboratory in our investigations of clays suitable for bonding graphite for crucibles check those obtained by Mr. Booze. That is, we have found Laclede-Christy B-291, or No. 3.01 in Mr. Booze's paper, to be one of the best single bond clays considering all the data so far available. The English Dorset ball clay is also a good single clay. The writer knows of one manufacturer who had excellent results with this clay as a substitute for the Klingenberg clay.

In laboratory experiments on bond clays, the writer has secured results checking Mr. Booze's statement "that the more plastic clays require a higher proportion of tempering water than do the lean ones." Also, the writer has noted that the slaking test is subject to error and for clays having similar plasticity gives very little differentiation.

The point mentioned by Mr. Booze in his concluding paragraph regarding a blended mixture of clays to substitute for a single clay, is one that merits attention. At the present time, the writer has under investigation a series of blended clays using clay 3.01, or B-291, as a basis together with two Enid clays. The investigation has not been carried to a point where comparative data can be submitted. It is anticipated that from this series a mixed bond clay will be found that will have properties better than any single clay so far reported.

The writer hopes that the results of Mr. Booze's work will be carried further by making actual factory-size crucibles and using the same under standard foundry practice. In this way the Society would have the benefit of a technical investigation applied to an industrial problem and under conditions far different from those existing in the laboratory. This, however, would cover a long period of time but the data obtained should justify the expenditure to this end. Mr. Booze is to be congratulated for this piece of work and the Society is indeed fortunate to have it on record.

Mellon Institute of Industrial Research,
Pittsburgh, Pa.

NOTE ON THE USE OF MAGNESIA AS AN OPACIFIER.

By V. S. Schory, Tiffin, Ohio.

Introduction.

In connection with a rather extended investigation for the purpose of producing a satisfactory glaze for one-fire ware, it was found convenient to make a brief study of the opacifying properties of magnesia in high-fire glazes. In a number of cases the writer had observed mixtures containing considerable amounts of magnesia which fired to opaque enamel-like coats with good gloss. Mention is made in the Transactions[1] of the use of magnesia as an opacifier in stoneware glazes at cone 7. When the first attempts were made to secure similar effects in porcelain glazes at from cones 9 to 11, the results were not uniform. In some cases the resulting coat was a good opaque white; again, with no great change in temperature or glaze composition, the glaze would be transparent and of a dirty, greenish yellow cast. Some of the first indications were to the effect that the addition of small amounts of barium and zinc resulted in increased transparency.

Experimental.

In order to prove or disprove these indications, and to secure sufficient definite data to explain the erratic results which had been obtained, the following brief study was outlined. The work was made systematic within the limits of heat treatment available in the kilns used in regular factory practice. The following glazes were prepared:

No. 1.	No. 2.	No. 3.	
0.30	0.30	0.30 K_2O	
0.20	0.30	0.40 CaO	0.50 Al_2O_3 4.00 SiO_2
0.20	0.10	0.00 ZnO	
0.30	0.30	0.30 MgO	

[1] R. T. Stull, *Trans. Am. Ceram. Soc.*, 11, 605 (1909).

No. 4.	No. 5.	No. 6.			
0.25	0.25	0.25 K_2O			
0.25	0.35	0.45 CaO	}	0.40 Al_2O_3	} 3.30 SiO_2
0.20	0.10	0.00 ZnO			
0.30	0.30	0.30 MgO			

No. 7.			
0.30 K_2O			
0.70 CaO	}	0.50 Al_2O_3	} 4.00 SiO_2

Glaze No. 1 is a duplicate of an opaque glaze given by Stull in the Transactions.[1] Glaze No. 7 corresponds to the formula of cone 4 and was prepared for comparison with the other members.

The glaze slips were concentrated to the plastic state and formed into briquettes. The dried briquettes were fired in commercial kilns to cones 5, 7, 9 and 12.

Results.

Glaze No. 1.—At cone 5 this mixture was an over-fired porcelain but the briquettes were not badly deformed. The surfaces had a fairly good gloss. The fracture of the pieces was stony. The color was a fairly good white.

At cone 7 the briquettes had softened sufficiently to lose shape and flatten out considerably. The surface gloss was inferior to that at cone 5. The fracture was less stony and more glassy than at cone 5 but showed no marked development of vesicular structure. The color was not quite so white as at cone 5.

At cone 12 the briquettes had fused down to form level lakes of glass, having better gloss than at any of the lower temperatures. The fracture was glassy. Throughout the mass were innumerable small bubbles. The color had become yellowish and was much inferior to the color at cone 9. Apparently, the glass had lost its opacity except that due to the bubbles present. When applied as a glaze coating no opacity was noticeable.

Glazes Nos. 2 to 6.—Briquettes of glazes Nos. 2 to 6, fired to the same temperatures, formed series similar to that of glaze No. 1. Briquettes of glazes Nos. 1 and 4 developed gloss at cone 5, becoming inferior at cones 7 and 9. The other numbers developed gloss by cone 7, becoming inferior at cone 9. At cone

[1] *Loc. cit.*, p. 612.

12 all members were fused to lakes of glass with better gloss than at any of the lower temperatures.

No considerable development of vesicular structure was observed in any glaze at cone 9, but at cone 12 innumerable small bubbles occurred throughout the mass in every case. In glazes Nos. 4, 5 and 6 the bubbles were larger than in glazes Nos. 1, 2 and 3.

Each mixture was a good white at the stage where it was a porcelain sufficiently overfired to develop good gloss. With increased heat treatment and softening of the briquettes there was a corresponding loss of color. At cone 12 all had become yellowish.

The loss of opacity was approximately concurrent with the development of the yellowish color. The more fusible members were only partially opaque at cone 9 and at cone 12 no opacity was evident in any of the fusions except that due to the bubbles present.

Glaze No. 7.—Briquettes of glaze No. 7 were porcelains slightly overfired at cone 5 and with glossy surfaces and rounded corners at cone 7. At cone 9 the briquettes had lost shape and were much inferior in color and opacity to the members containing magnesia. At cone 12, glaze No. 7 was similar to glazes Nos. 1, 2 and 3.

The effects of magnesia as noted in glaze No. 3 were the maintenance of good color and opacity to a higher temperature than obtained in the case of glaze No. 7. The difference between glazes No. 3 and 7 were not so marked at cones 5 and 7, but at cone 9 glaze No. 3 was much the superior in color and opacity. The action of zinc was primarily to promote early fusion. It had no marked effect on color in these glazes.

Comparison with Commercial Glazes.

For further comparison with these mixtures, briquettes were made from two commercial glazes, a stoneware glaze maturing at cone 8 and a porcelain glaze maturing at cone 10. At cone 5 the briquette of stoneware glaze had fused down to a viscous fluid state and showed incipient vesicular structure. The surface was dull. At cone 7 the fluidity, vesicular structure and gloss had increased. At cone 9 the mass had lost opacity and contained many bubbles. When applied as a glaze coating and

fired to cone 8 the glaze was bright but retained some small bubbles.

Briquettes of the porcelain glaze fused down to a viscous fluid state with some vesicular structure at cone 5. The surface gloss was fairly good at cone 5 and improved with increase in heat treatment to cones 7 and 9. At cone 9 the fusions contained a number of rather large bubbles. When applied to ware in the usual manner this glaze cleared satisfactorily at cone 9.

It was noted that glaze No. 1, for example, lost gloss from cone 5 to cone 7, but that the stoneware glaze, though more fusible, was dull at cone 5 and developed better gloss with increase in heat treatment. The porcelain glaze at cone 5 developed gloss which improved at higher temperatures.

Conclusions.

From the data it appears that glazes Nos. 1 to 6, when fired to form opaque, glossy coats, are really in the condition of over-fired porcelains and may be more accurately designated as glossy, vitreous slips. It was found also that zinc and barium did not increase the transparency of the coats except as they increased the fusibility.

DIRECT VOLUME-DETERMINATION IN A VOLUMETER OF THE PYCNOMETER TYPE.

By J. B. Shaw.

In a recent article, H. G. Schurecht[1] illustrates a pycnometer which he has used in measuring the volumes of clay test pieces. He offers considerable criticism, which is certainly somewhat justified, of the Seger volumeter, because of its complex construction, fragility, inaccuracy, and slow operation. There is a real need for some simpler type of volumeter and as the writer has been using one similar to that illustrated by Schurecht, but differing in one essential, it was deemed worth while to give a description at this time.

The volumeter (Fig. 1) does not differ materially from Schurecht's in general dimensions; in fact, it was purposely made to conform with his since the size shown is very adaptable for ceramic work. The volumeter differs essentially from the pycnometer in the size of the orifice in the glass stopper. The tube in the stopper in this volumeter has an internal diameter of $^1/_4''$ so as to permit the liquid to flow into the bottle from a burette.

Following is the procedure in measuring the volume of a briquette:

(1) Saturate the briquette with liquid (kerosene if green, water if burned).

(2) Place the briquette in the volumeter.

(3) Fill the volumeter to the mark.

(4) Remove stopper and briquette.

(5) Replace stopper in same position as original.

(6) Run liquid from the burette through the tube in stopper until the bottle is filled to the mark.

(7) Read the volume directly from burette.

Schurecht's claim to accuracy must be admitted. His claim to speed and cheapness are doubted. One who makes two weighings, accurate to 0.01 or 0.05 g., and four mathematical calcula-

[1] *J. Am. Ceram. Soc.*, 1, 556 (1918).

tions, each involving four figures, in two minutes, will work more rapidly than the average man and probably too fast for accuracy. For weighing pieces of 300 to 400 grams mass, a balance sensitive to 0.01 g. is more costly than a Seger volumeter. There is no desire on the part of the writer to detract in any way from the real merits of a volumeter of the pycnometer type; it is recognized as having many points in its favor and is certainly to be preferred to the Seger volumeter. Its use, however, seems to involve refinements not required in making other tests on the same kind of test pieces.

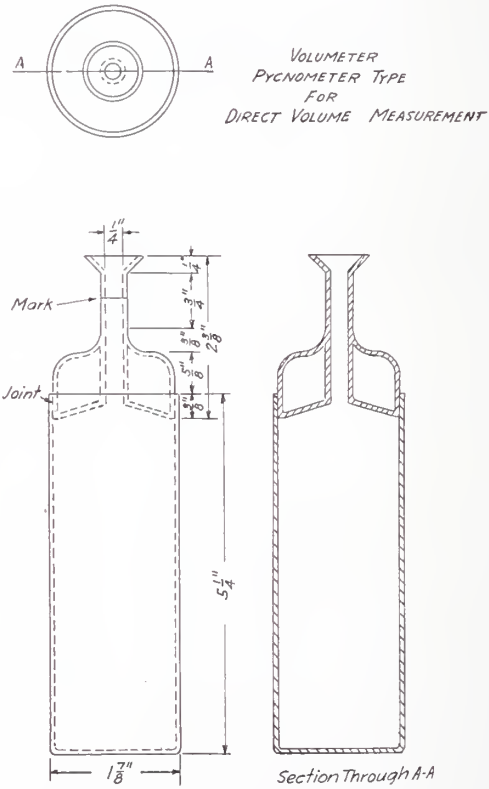


FIG. 1.

The volumeter illustrated required only a burette in addition to the pycnometer in making volume determinations. The

burette can be read accurately to 0.1 cc. and estimated to 0.05 cc.

The experimental error in manipulating this apparatus will approximate about 0.1 cc., which in the average determination would represent an error of about 0.30 per cent. It is doubtful if greater accuracy is required in work of this kind.

As the Standards Committee of the American Ceramic Society has recommended that briquettes be weighed accurately to 0.1 g., it would be useless to attempt to measure volumes to a greater degree of accuracy.

The chief points of merit claimed for this volumeter are: (1) speed, (2) simplicity, (3) low cost, (4) sufficient accuracy.

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COMMUNICATED DISCUSSIONS.

H. G. SCHURECHT: Mr. Shaw states in his paper that it is impossible to determine the volume of a briquette in 2 minutes with the pycnometer-volumeter described by me in this JOURNAL.[1] When the weighings and calculations are made in the customary manner, Mr. Shaw's assumption is correct, but by making the balance read automatically for fractional gram intervals, and by using tables for volume calculations, such determinations were made in the Bureau of Mines laboratories in an average of one and one-half minutes, when weighing a large number of test pieces. These methods were as follows:

Making the Balance Read Automatically.—This may be done with most chemical balances in the manner shown in Fig. 2. By attaching a weight (an ordinary screw pinchcock may be used) to the needle indicator, the moment W × A becomes greater. This may be so adjusted by moving the weight up or down on the needle, so that when the needle is 10 major divisions to the right or left of the zero point, it may be brought to zero by adding a one-gram weight to the opposite pan. This makes each intermediate division read 0.1 gram and with the balance as used the weight could easily be estimated to 0.025 gram by noting the position of the indicator. Hence, it is only necessary

[1] *J. Am. Ceram. Soc.*, 1, 556 (1918).

to place a weight, within one gram of that of the briquette, on the pan and the weight above or below that of the briquette may be read automatically, thus saving the time otherwise necessary in handling the smaller weights. For example: if a briquette weighs 40.75 grams, place the briquette on left pan, a 40-gram weight on the right pan, release the balance slowly (thus preventing vibrations of the needle) and the needle will come to rest at 7.5 divisions to the right of center.

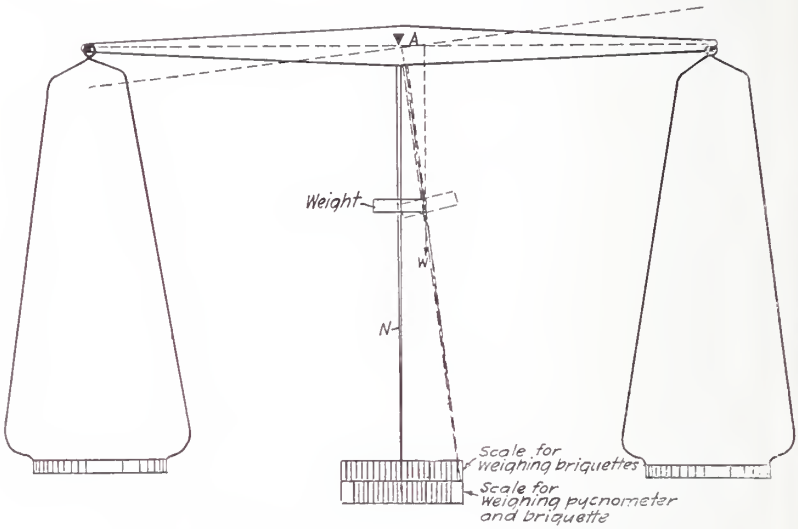

FIG. 2.—Balance with automatic weighing attachment.

It was found that for heavier weights, above 100 grams, as is necessary when weighing the pycnometer-volumeter, the indicator did not swing quite as far to the right or left upon the addition of a gram weight, this probably being due to the increased friction caused by increased weight. It was therefore necessary to use another scale, calibrated for use with the pycnometer, which was pasted to the lower half of the original scale (see Fig. 2). Where the briquettes are about the same size, the time of weighing is reduced about $66^2/_3$ per cent by using the automatic attachment.

Obtaining Volumes from Tables.—The calculations involved

for determining the volume with a pycnometer-volumeter are as follows:

$$V = \frac{W - W_1 + B}{S} = W - \frac{(W_1 - B)}{S} \text{ in which}$$

V = volume in cc.
W = weight of pycnometer plus liquid.
W_1 = weight of pycnometer plus liquid plus briquette.
B = weight of saturated briquette.
S = specific gravity of liquid.

The first step is to obtain $W_1 - B$, then $W - (W_1 - B)$, and finally

$$W - \frac{(W_1 - B)}{S} = V.$$

In preparing the tables it is necessary to obtain sufficient data to cover the upper and lower limits of these values, which vary with the size of briquette and size of volumeter. The following pre-calculated tables may then be prepared, varying the small variable from left to right and the large variable from top to bottom as indicated. For example:

$B = 44.85$
$W_1 = 142.85$
$W_1 = 168.50$
$S = 0.816$

To determine $(W_1 - B)$, run down the left hand column of Table 1 to 44.85 and then follow this line to the right until under 168.50, which gives the pre-calculated difference, 123.65. Since the values for W_1 varied over a comparatively large range of numbers it was found impossible to arrange all of the data on one table so they were separated into a number of tables which were bound together and indexed on the right hand side. For example: one table contains all numbers between 168.00 – 168.95, the next 169.00 –169.95, etc.

With 123.65 and 142.85 (W_1), the value $W - (W_1 - B)$ is obtained in a similar manner from Table 2 and found to be 19.20.

With 19.20 and 0.816 (S), the value of V is then obtained from Table 3 and found to be 23.53.

When determining the volumes of a large number of test pieces which are approximately the same size, as is generally the case when testing clays, the use of the automatic weighing attachment and tables for determining volumes is recommended because of the time saved, since the accuracy in weighing is far below that of the other experimental errors.

TABLE 1.—To determine (W_1 — B).

W_1. ⟶

B ↓	168.00	168.05	168.30	186.35	168.40	168.40	168.50	168.55
44.60	123.40	123.45	123.70	123.75	123.80	123.85	123.90	123.95
.65	.35	.40	.65	.70	.75	.80	.85	.90
.70	.30	.35	.60	.65	.70	.75	.80	.85
.75	.25	.30	.55	.60	.65	.70	.75	.80
.80	.20	.25	.50	.55	.60	.65	.70	.75
.85	.15	.20	.45	.50	.55	.60	.65	.70
.90	.10	.15	.40	.45	.50	.55	.60	.65
.95	.05	.10	.35	.40	.45	.50	.55	.60
45.00	.00	.05	.30	.35	.40	.45	.50	.55

TABLE 2.—To determine W — (W_1 — B).

W. ⟶

(W_1 — B) ↓	142.60	142.65	142.70	142.75	142.80	142.85	142.90	142.95
123.50	19.10	19.15	19.20	19.25	19.30	19.35	19.40	.45
.55	.05	.10	.15	.20	.25	.30	.35	.40
.60	.00	.05	.10	.15	.20	.25	.30	.35
.65	18.95	.00	.05	.10	.15	.20	.25	.30
.70	.90	18.95	.00	.05	.10	.15	.20	.25
.85	.85	.90	18.95	.00	.05	.10	.15	.20
.80	.80	.85	.90	18.95	.00	.05	.10	.15
.85	.75	.80	.85	.90	18.95	.00	.05	.10
.90	.70	.75	.80	.85	.90	18.95	.00	.05
.95	.65	.70	.75	.80	.85	.90	18.95	.00

TABLE 3.

To determine $\frac{W - (W_1 - B)}{S} = V.$

W — (W₁ — B) ↑	S. → 0.814	0.815	0.816	0.817	0.818	0.819
		23.00				23.20
19.00	23.34	23.31	23.28	23.26	23.23	23.20
.05	.40	.37	.34	.32	.29	.26
.00	.46	.43	.40	.38	.35	.32
.15	.52	.49	.46	.44	.41	.38
.20	.60	.56	.53	.51	.47	.44
.25	.65	.62	.59	.57	.54	.51
.30	.71	.68	.65	.63		
.35	.77	.74	.71			
.40	.83	.80	.76			
.45	.89	.86	.83			

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J. B. SHAW: There seems little doubt that by the use of the tables illustrated by Mr. Schurecht the time of making volume determinations in the pycnometer-volumeter would be very materially reduced. It seems obvious, however, that the method of reading volumes direct is bound to require less time than any method requiring weighing—regardless of how many short-cuts are used.

It is doubtful if the slightly higher degree of accuracy obtained by the use of this elaborate set of tables and expensive balances will justify their use in the average ceramic laboratory. Where great accuracy is desired, this method is probably the best yet devised, but for ordinary purposes the volumeter, reading volumes directly, is sufficiently accurate and certainly decidedly cheaper.

AN AMERICAN PORCELAIN CONTAINING NO FREE SILICA.

By Arthur S. Watts.

The following is one of a series of studies conducted with the object of producing a practical porcelain body which is free from the faults due to the presence of free silica. The chief of these faults is supersensitiveness to sudden change of temperature. Second only to this fault is the tendency to excessive warping The mode of procedure in this study was as follows:

A standard porcelain body was separated into its two essentials: (*a*) plastics, including all clays, and (*b*) non-plastics, including all feldspar, whiting, and flint. The non-plastic portion was calcined at a temperature just sufficient to produce a pyrochemical action between the feldspar and whiting and to allow them to act on the flint. Such a calcine, however, must not reach the glossy stage as it would be too difficult to pulverize. The calcine or semi-fusion was chilled by plunging while hot into cold water and the friable mass resulting was then pulverized in a ball mill. The pulverized calcine and the clays were then mixed in such proportions as to produce a porcelain having the same chemical composition as the normal porcelain with which it was compared. For this study the following bodies were prepared:

	Body I. Per cent.	Body J. Per cent.	
Canadian feldspar	20	20	Calcined at cone 3.
Whiting	2	2	
Flint	33	33	
Tennessee ball clay No. 7	10	10	
Florida kaolin	10	10	
Edgar Georgia kaolin	25	25	

The calcine vitrified to a dense, opaque mass at cone 3. A microscopic study disclosed, however, that all of the flint had not dissolved but that about 10 to 12 per cent free flint remained. The calcine, after removal from the kiln, was reheated and plunged

into cold water, resulting in the shattering of the mass into small fragments which were ground in about 5 hours into a powder which passed a 200-mesh sieve with 2 per cent residue.

The two bodies, I and J, were made up and fired to cones 7 and 10.

At cone 7, Body I developed a cream color and an absorption of 4.4 per cent, while Body J developed a good white color and had a vitreous structure, the absorption being 2.12 per cent. At cone 10, Body I developed a pale-cream color and an absorption of 0.7 per cent, while Body J was blue-white in color and non-absorbent. The color and absorption were thus greatly improved by the calcination of the non-plastics.

The two bodies were then tested for resistance to sudden temperature changes with the following results: Body J, even though of denser structure, possessed approximately three times the resistance to sudden temperature change displayed by Body I—prepared exclusively from raw materials.

Conclusions.

By calcining or semi-fusing the fluxes and flint prior to incorporation in a porcelain body, the maturing point can be lowered approximately two cones, the color vastly improved, and the resistance to sudden temperature changes improved approximately 200 per cent.

The objection to such a method of manufacture is the extra cost of preparing such a calcine. To offset this we have the saving in kiln maintenance and the fuel saving from the lowering of the maturing temperature of the bisque fire. The degree of pulverization necessary in the feldspar used could doubtless be reduced somewhat which would also mean some saving.

Another possibility which is apparent from this study is the reduction in the amount of flux required in case a lowering of the firing temperature is not desired.

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A SIMPLIFIED APPARATUS FOR THE DETERMINATION OF AIR IN CLAY.

By H. Spurrier.

During the last year the determination of air in pugged clay has assumed increasing importance. It became desirable to simplify the determination so that it might be performed as a factory routine test by entirely unskilled help.

All efforts failed to simplify the previously described apparatus[1] sufficiently to make it a hardy, accurate, and at the same time, safe piece of apparatus to put into factory hands; consequently, it was determined to abandon any corrections, and to simply disengage the gases occluded in the clay by the use of air-free and nearly boiling water, and to see how nearly these results agreed with those obtained by the more elaborate apparatus. Accordingly, a broken burette was cut off evenly and fused to the shortened tail-piece of a large "carbon funnel" which was placed, inverted, in a liter beaker (Fig. 1).

The clay blanks are prepared of such a size that they may easily be placed in the bell of the funnel. 800 cc. of water is placed in the beaker and boiled for some time. The funnel is inserted into the beaker, the stop-cock at the upper end being open. By means of a short piece of rubber tubing fitted to the inverted tip of the burette cock, the water is sucked up till it fills the apparatus to the bore of the cock, thus moving the air entirely from the apparatus. The bell is now raised high enough so that it may be rapidly lowered over the blank which is quickly placed in the vessel of water. The evolution of the air goes on concurrently with the slaking down of the clay blank.

The separation of the air enmeshed in the finely disintegrated clay is greatly facilitated by a rapid gyration of small amplitude,

[1] *J. Am. Ceram. Soc.*, 1, 710–715 (1918).

coupled with a slight up- and down-motion; this may be accomplished without the escape of any air from the apparatus. After a few minutes all of the air is collected in the upper portion of the burette and the reading is taken. The froth that collects

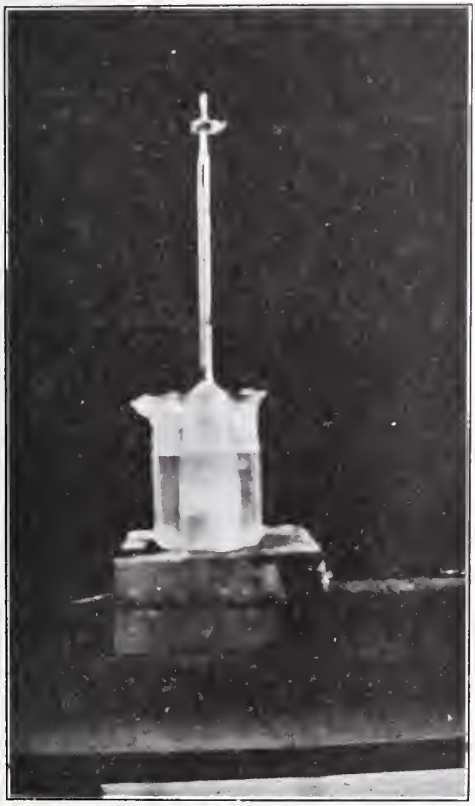


FIG. 1.

above the water line is neglected. The percentage volume of air is calculated on the basis of the volume of the blank used.

Undoubtedly errors are involved in this procedure, but it was foreseen that they would be of a compensatory nature, as proven by the results.

So successful were the results of the tests that a suitably designed one-piece apparatus was ordered from the local glass-blower. The photograph (Fig. 1) shows the apparatus as used and the drawing (Fig. 2) shows the dimensions found suitable for testing our clay blanks.

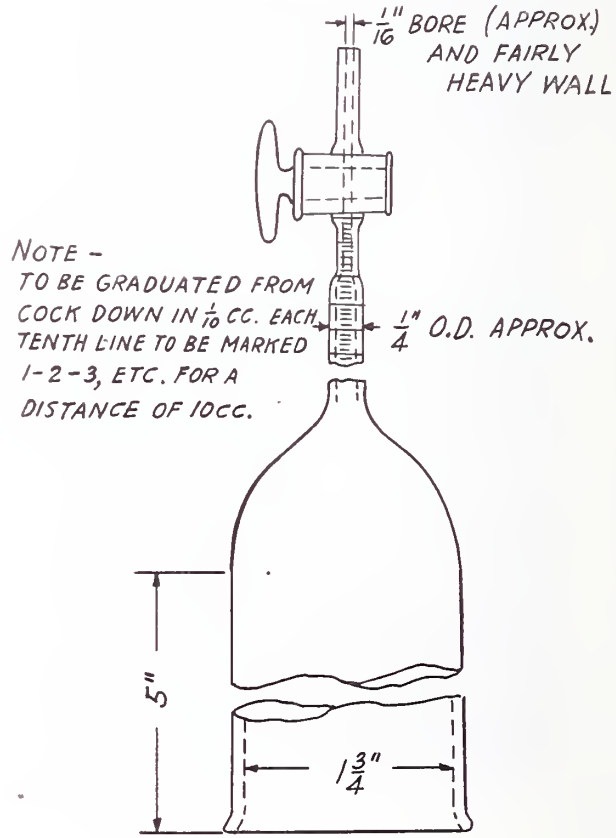


FIG. 2.

Routine results have amply justified the expenditure of time involved, as anyone of average intelligence may be instructed in the use of the apparatus in one-half hour and can make a large number of determinations concurrently. For convenience, and

to avoid all calculations, charts were prepared showing the percentage of air by volume calculated on the various sizes of blanks in use, so that the operator reads off the volume of air separated and refers to his table for its percentage—which is duly recorded in the process-inspection report.

JEFFERY-DEWITT COMPANY,
DETROIT, MICHIGAN.

CERAMIC ABSTRACTS.

Abstractors.

E. D. Elston — J. S. Laird
Earle N. Jessop — G. E. Middleton
Chas. H. Kerr — H. G. Schurecht
F. A. Kirkpatrick — Chester Treischel

Microscopic examination of clays. R. E. Somers. *J. Wash. Acad. Sci.* **9**, 113–126, (1919).—The investigations as stated by Somers are not intended to be exhaustive. Nevertheless the results are well worth some attention. The microscopic examination was made to determine the mineral content and in some cases petrographic study was made of the burned samples in order to ascertain, if possible, what changes occurred during the burning. By means of a table the author has contrived to place before the reader in a more graphic form the mineral content of the unburned clays together with a comparative measure of the relative abundance of the more important minerals in each sample. Such a method is greatly benefited by the author's brief description regarding the identification of the various minerals.

Of special interest is the fact that Somers states that hydromica is apparently quite abundant in some of the clays examined. Further, "the hydromica has single and double refractions higher than those of kaolinite, yet not so high as muscovite or sericite" and that "the degree of these refractions varies in different clays." Therefore, Somers assumes "that there is an isomorphous gradation between sericite and kaolinite, with a gradual loss of potash and addition of water, and, in weathering products such as these, hydromica represents a transition stage of weathering toward kaolinite as a final product." In addition to abundance of hydromica in some clays as shown by petrographic examination, Somers thinks that since many kaolins show by chemical analysis a small percentage of potash, the latter may be regarded as belonging to the mica, inasmuch as feldspar seems to be very scarce. Halloysite was found in but two of the many clays examined while rutile in some amount was contained in practically all of them. Thin sections disclosed the presence in some of the clays of tourmaline, epidote, zircon, titanite and diaspore.

Some of the clays were burned and thin sections of the fired material were examined with the results that the following facts were noted: Quartz grains were more prominent in the fired product than in the raw clay—due to the fusing of the hydrous aluminum silicates into a fine-grained mass holding the quartz. In some specimens a fluxing action appeared to have occurred between the fine-grained material and the silica, with resulting corrosion of the quartz. This, according to the author, was comparatively rare. Since

the hydromica, on heating to 1150° C, either disappears or loses most of its interference colors, it would seem that it may furnish some of the flux for the clay. Tourmaline and probably epidote disappear at 1150° C, but rutile zircon and probably titanite apparently are persistent at 1300° C.

It is interesting to note that in a Florida white clay fired at 1300° C sillimanite developed from the large flakes of kaolinite or low-grade hydromica as indicated by one flake containing both minerals. The actual cause for its development, however, is unknown. Other clays containing similar micaceous particles did not show sillimanite when burned, although they were fired at the same time as the Florida sample. In Mr. Somer's opinion this development of sillimanite by burning and its presence in porcelain may explain the cause of double refraction produced in the ground mass of some clays by burning to higher temperatures. The tables given in the article also show the texture of clay, the relative abundance of the important constituents, and the porosity after burning. E. D. ELSTON.

Baked shale and slag formed by the burning of coal beds. G. SHERBURNE ROGERS. U. S. Geol. Surv., *Prof. Paper* 108-A, (1917).—The effects produced by the burning of coal beds have been observed particularly in portions of Montana. Mr. Rogers is of the opinion that ignition, which in some instances may be due to any one of a series of causes, such as lightning, forest or prairie fires, or to the agency of man, appears in most cases to be due to spontaneous combustion. The appearance of the rocks overlying a burned coal bed varies, ranging from reddened and only slightly hardened condition of the shales or sandstone through glassy slag to gray medium-grained rock. These differences seem to be due chiefly to the degree of heat to which the material has been subjected. The combustion, according to Mr. Rogers, apparently starts at the surface, spreading first along the outcrop. As the coal burns out the overburden generally caves, and large fissures may result. During this stage of the burning the overlying strata are not greatly affected, owing to the dissipation of the heat. Clay partings, however, may be baked to form thin, red bands. With the progressive burning back from the outcrop the overlying rocks are more strongly affected. The underlying rocks are only slightly changed, if at all. From the burning beds upward there appears to be a gradual transition. Near the burning bed and extending upward a few feet the rocks may be partly fused. Above they are in general only baked and further up only slightly reddened. Much of the shale is hardened or vitrified, although the sandy material may be only slightly affected. The reddening of the rock, according to Rogers, is due to moderate heat and implies no extensive chemical change.

In general, the shale or sandstone in more or less baked condition forms the larger part of the metamorphosed materials. The shales lost their original texture, becoming hard and massive. Conchoidal or splintery fracture is not uncommon and many specimens resemble jasper. Commonly the rock is mottled, probably due to higher temperature and the colors produced by irregular oxidation of iron. The vitrified portions display in thin sections

the presence of hematite and magnetite, the former in the red areas while the latter occurs in green and black areas. The rock is rough and vesicular, somewhat similar to some types of brick. Notable among the chemical changes, is dehydration under conditions favorable to oxidation. This affects chiefly the ferrous iron compounds, which are converted to hematite with reddening of the rock. E. D. ELSTON.

"Clay-working industries and building operations in the larger cities in 1917." JEFFERSON MIDDLETON. U. S. Geol. Surv., *Min. Res. of U. S.*, Part II, 28(1917).—For general conditions of the clay industries, including tables showing production, this publication, though not dealing with specific location and detailed accounts, presents much material commercially interesting to clay workers. E. D. ELSTON.

Electricity in the ceramic arts. J. P. ALEXANDER. *Gen. Elec. Rev.*, 22, No. 2 (Feb., 1919).—"Until recent years, electricity had not been introduced into pottery and tile-making factories for the reason that it had been generally supposed that a steam engine, by reason of the excess power it could produce over its normal rating, was the only driver that could start up the machinery after the clay had settled in the mixing tubs overnight. Recent electrical installations have shown the fallacy of this idea, and have proved to ceramic engineers that the electric motor can duplicate any of the engine drives." The writer proceeds to detailed descriptions of the various processes employed by the ceramic industry and outlines the increasing service afforded by electricity in this field. C. TREISCHEL.

Crystalloids against colloids in the theory of cements. H. LE CHATELIER. *Trans. Faraday Soc.*, 14, 8 (1919).—The colloidal theory of the hardening of cements is devoid of sense. In a similar way it used to be said that bodies react because they have affinity for each other. The explanation seems to come down to this: cements harden because they have the property of hardening. Let us define the word "colloid" if possible. Graham found a certain few distinctions between "colloids" and "crystalloids." Zsigmondy showed that the collidal solutions were merely suspensions of very finely divided, rigorously insoluble matter. All very minute insoluble bodies give such colloidal suspensions. But is there cause to give a particular name to bodies, the only singular properties of which so far are insolubility and fineness? These very minute bodies possess, however, one quite special and very important property, which entitles them to be classed separately, that is the property of absorbing the soluble bodies which are held in solution by the liquid with which they are wetted. This property manifests itself also in porous bodies, such as blood carbon and wood charcoal. Hence, from this point of view, colloids and porous bodies must be classed together. This absorptive power is a particular case of much more general properties which all very fine bodies possess. These particular properties are under the sway of surface tension. All chemistry is only a chapter on energy; but there is one form of energy of which chemistry does not speak as a rule, that is

surface energy, which is measured by the product of the surface tension and of the free surface of the body. The surface energy of water, for example, in the form of a sphere weighing 18 gm. is 6.3×10^{-8} kg. calories. If this sphere were divided into globules, each 10^{-6} mm. in diameter, the surface energy would be equivalent to 2 calories, or of the order of magnitude of the quantity of heat which water evolves in its various chemical reactions. Certain indirect considerations lead us to admit that surface tension of solids in suspension may become a hundred times greater than that of water. However, we have not yet considered the solubility of the bodies in question. A solid crystal ground to very fine grains will have all the properties of colloids until it is brought into contact with a liquid which can partially dissolve it. There will result an increase in the solubility of the fine crystals and large crystals will soon separate out. This has been verified for the very fine ppts. of CaC_2O_4 and $BaSO_4$. We come now to hydraulic mortars. When plaster of paris has set, we can never discern any crystals in the material, even under the strongest magnification of the microscope. That does not prove that no crystals are formed; perhaps they are too small to be recognized. They do form in several weeks if the plaster is left to itself or if the plaster is gauged with alcoholic water. The calcium aluminates, which play an important part in the hardening of rapidly setting cements, are hydrated at the same rate as the plaster of paris, and they give likewise a whitish mass in which crystals are not discernible. If instead of gauging the aluminate with a deficiency of water we put a few isolated grains into a large volume of water, we observe that the grains soon become surrounded by long crystals of hydrated aluminate, which are always easily visible under the microscope and can sometimes be seen directly by the eye. The microscope has never enabled us to see hydrated calcium silicate ($CaOSiO_2 \times H_2O$). Its crystals are certainly very minute. Numerous analogies prevent us, however, from denying their existence. Crystals of barium silicate can always be seen with the aid of a lens, sometimes even with the naked eye. Hence, we may say by analogy that calcium silicate crystals will form, although they are, on account of their smaller solubility, too minute to be discernible. It is not impossible that, initially, the dimensions of these crystals are of colloidal order. In the course of time the dimensions of the crystals must increase considerably, as in all similar instances. The mechanical resistance does not diminish with time, quite to the contrary. Consequently, the colloidal state which was possible at the beginning has nothing to do with the hardening of the cement. The anhydrous compounds of cements, not being in chemical equilibrium when in contact with water, are more soluble than the same hydrated compounds. For this reason they give rise to the formation of a supersatd. solution from which the dehydrated compound may rapidly crystallize. The liquid, being no longer saturated, can redissolve more of the anhydrous compounds and the whole mass can thus crystallize progressively by passing through a stage of transitory dissolution in a limited volume of water, which, at any given moment, cannot maintain in solution more than a small portion of the total mass of salts present. The crystals which are deposited from the

supersaturated solutions always appear in the shape of extremely elongated needles, which have the tendency to group themselves in spherulites about the centers of crystallization. The entanglement of all these crystals is the only cause of the solidity of mass: they adhere by simple contact, as do all solid bodies when resting upon one another. Friction at the points of contact, even in the absence of all adhesion, suffices to produce great mechanical resistance, such as is found in all tissues and fabrics whose solidity has no other origin. Adhesion is increased and friction decreased by the growth of the crystals. A material built up of very wide strips could be very solid if the strips were glued together at their contact surface.

F. A. KIRKPATRICK.

ethods of fuel conservation in cement mill operations. ERNEST ASHTON. *Concrete Cement Mill Section*, **13**, 45 (1918).—(1) The fusion temperature of the cement may often be lowered by addition of the proper ingredients. (2) Uniformity of size of particle is more desirable than extreme fineness of part of the material. The fine material fuses sooner than the coarse, causing the formation of balls which do not burn properly. (3) The rate of feeding and the speed of the kiln should be so regulated that the amount of raw material will be the maximum that can be carried without cooling the kiln when the greatest amount of coal that can be burned with complete combustion is being blown into the kiln. (4) The Bureau of Mines' recommendations in regard to storage of coal should be carried out as closely as possible. (5) The coal should be ground uniformly and finely. (6) Utilization of stack and clinker heat should be more thoroughly studied and the necessary installation of equipment made. The kiln of the future should be almost air-sealed at the front, with preheated air to aid combustion, and waste heat boilers should be used. (7) There are described economical methods of oil burning for cement kilns. (8) Power plant operation should be as thoroughly controlled as it is in any modern industrial plant.

F. A. KIRKPATRICK.

Furnaces for obtaining high temperatures. A. BIGOT. *Chemie et Industrie*, **2**, 27–36 (1919).—A laboratory furnace in which specimens of considerable size can be heated to 1750° C has been developed by modifying the design of the Perrot down-draft pot furnace. The iron pipes used in the latter for carrying off the products of combustion and for introducing the secondary air in such a way that part of the heat of the waste gases is recuperated have been replaced by a refractory tube carried in a well insulated refractory flue, so that the air is highly preheated. The furnace proper is lined with fused alumina and is very carefully insulated. With a capacity of about 2 cu. ft. it requires about 300 cu. ft. of gas per hour to heat it to 1750° C in 6 hours. The author has also developed industrial continuous kilns in which he obtains great economy by making use of the same principles of insulation and recuperation. In these the cars with iron wheels used in other tunnel kilns have been replaced by conveyors made of refractory slabs which

roll on refractory balls set in the refractory slabs which line the bottom of the kiln. These refractories, made of special composition not given in the article, have at 1100° C a crushing strength of about 20,000 pounds per sq. in. For kiln temperatures above 1100° C, at which the refractories would become much weaker, the movable parts are sunk in a trench to protect them from overheating. The mass of material to be heated is much less than it is when ordinary cars are used. Firing, preheating and cooling are carried on as in other types of tunnel kilns. Kilns are 100–150 ft. long, and have an output of 15–25 tons of clay-ware per day. The kilns are being used for the firing of various kinds of ware, including silica and other refractories. The maximum length of firing is 5 days. The following comparative data are given:

	Percent of heat utilized at		
Type of kiln.	1000° C.	1250° C.	1400° C.
Intermittent	20%	....	10%
Hofmann	40%	40%	....
Car tunnel kiln	80%	50%	40%
Bigot tunnel kiln	....	78%	70%

Ware.	Temp of firing.	Fuel consumption per ton product.
Stoneware	1250° C	100 lbs.
Porcelain	1350° C	122 lbs.
Bauxite, chrome	1450° C	150 lbs.
Magnesia	1530° C	210 lbs.
Silica	1530° C (longer fire)	260 lbs.

J. S. Laird.

CERAMIC PATENTS.

G. E. Middleton, *Abstractor.*

Brick or tile dies. C. H. Brisbin. U. S. 1,301,095, Apr. 22, 1919.—The lower die of the set consists of two plates normally spaced apart by spiral springs. There are cores on the lower plate projecting through apertures on the upper, the ends of the cores being held normally flush with the upper surface. Under compression the plates contact and the cores project above the surface of the upper plate, forming depressions in the brick or tile. When the pressure is removed, the cores recede and the brick or tile may be slid from the die surface.

Apparatus for manufacturing hollow glassware. L. N. Bruner. U. S. 1,301,378, Apr. 22, 1919. A machine for molding short, glass tubes or cylinders. The article is formed by centrifugal force in a rotating mold provided with a cup which works up and down therein pistonwise. The cup is raised to the top of the mold to receive the glass and is then lowered to the bottom thereof, the mold being rapidly rotated, thus causing the glass to spread evenly up the inside of the mold.

Apparatus for making bricks. H. W. B. GRAHAM. U. S. 1,301,685, Apr. 22, 1919. A soft mud brick machine in which the molds are filled, dumped, sanded and returned and the brick carried off, mechanically.

Method of and apparatus for forming glass tubes. K. KUEPPERS. U. S. 1,301,714, Apr. 22, 1919. Glass tubes of exact inner form are made by mounting the tubes on a mandrel of the desired shape, heating to soften, and pressing the tubes around the mandrel in successive stages lengthwise thereof.

Method of drawing hollow glass articles. O. A. WELLS. U. S. 1,301,771, April 22, 1919. Hollow glass articles are drawn by a process involving the use of a heated bait having a temperature below that at which glass will fuse thereto, but high enough to have substantially the same radial contraction during the draw as that which occurs in the novel of the glass article being drawn.

Brick for brick presses. C. W. WILKINSON. U. S. 1,302,113, Apr. 29, 1919. A brake band extends about the periphery of the rotary table of a brick press, its function being automatically to stop the rotary table and hold it after each movement and during the pressing operation, thus preventing the so-called back lashing or rearward movement of the table.

Drawing-pot for glass. R. L. AND F. J. FRINK. U. S. 1,302,149, April 29, 1919. An apparatus designed for melting and distributing or draining off the solidified glass which accumulates in drawing pots to which the metal is periodically introduced by dipping. Two pots are mounted, one at either end of a horizontally rotatable frame, and while a cylinder is being drawn from one, the other, tilted to a vertical position, is being subjected to an ignited gas blast which melts the accumulated glass and allows it to drain off. There are also means for introducing air at the base of the rising cylinder and through the bottom of the drawing-pot.

Press for lavatories. D. W. MCNEIL. U. S. 1,302,191, April 29, 1919. A fluid-operated dry-press designed to mold lavatories of the type having an integrally formed back or splash board, a rolled rim, an integrally formed apron, and a hidden overflow passage.

Structural composition. E. R. TAYLOR AND I. FLEXNER. U. S. 1,302,452, Apr. 29, 1919. A composition adapted for use in constructing buildings and for similar purposes. It is said to be dense, compact, soundproof, of substantial transverse strength and of a specific gravity about one-half that of ordinary concrete. Nails can be readily driven into it. The composition is variable within limits, an example being Portland cement 96 parts, mineral fiber, such as asbestos 24 parts, sawdust 6 parts, sand 8 parts.

DIVISIONS AND SECTIONS

Refractories Division.

A meeting of the Refractories Division of the American Ceramic Society will be held at Niagara Falls, August 4th, in connection with the Summer meeting of the Society. It is very important that as full an attendance as possible be had, owing to the fact that several important matters dealing with the future work of the Division will be discussed.

(Signed) A. V. BLEININGER,
Chairman.

Northern Ohio Section.

A meeting of the Northern Ohio Section of the American Ceramic Society was held at "Willoughbeach," Cleveland, O., on Monday, June 23, 1919.

After luncheon at the Hotel Cleveland, the party proceeded by trolley to "Willoughbeach." A business session was first held at which matters pertaining to the constitution of the Section and the arrangements for the Summer meeting of the American Ceramic Society, in Buffalo and Cleveland the first week in August, were discussed.

At the request of certain members of the Section, instead of the usual series of formal papers, the meeting was devoted mainly to a "Symposium on Fuels," particularly substitutes for natural gas. This included a discussion of the relative merits of coal, powdered coal, producer-gas, oil, etc. As a part of the discussion, Mr. W. T. Dean, President, General Combustion Co., presented an interesting paper on "Fuel Oil and Methods of Burning Same."

Following the meeting the members enjoyed the bathing and other facilities for amusement at the beach.

B. A. RICE, *Secretary.*

Eastern Section.

The Summer meeting of the New Jersey Clay Workers' Association and Eastern Section of the American Ceramic Society

was held at the Trenton Country Club, Trenton, N. J., on Tuesday, June 24, 1919.

At the opening session in the morning the charter granted by the American Ceramic Society to the Eastern Section was accepted and the following officers were elected to serve for the balance of the year:

Councilor, Charles A. Bloomfield, Metuchen, N. J.

Chairman, Charles H. Cook, Cook Pottery Co., Trenton, N. J.

Vice-Chairman, Abel Hansen, Fords Porcelain Wks., Perth Amboy, N. J.

Secretary-Treasurer, George H. Brown, Rutgers College, New Brunswick, N. J.

The following program was then presented:

Address by the Chairman, Charles H. Cook.

"Methods of Ceramic Kiln Control," G. H. Brown, New Brunswick, N. J.

"Some Data on the Development of Terra Cotta Glazes," by E. C. Hill, Conkling-Armstrong Terra Cotta Co., Philadelphia, Pa.

"A Plea for the Use of Precise Data in Ceramics," by A. V. Bleininger, Bureau of Standards, Pittsburgh, Pa.

"Ceramic Kiln Insulation," by P. A. Boeck, Celite Products Co., New York, N. Y.

"Some Aspects of Ball Mill Grinding," by Leslie Brown, Lenox, Inc., Trenton, N. J.

Luncheon was served at the Country Club during the intermission between the morning and afternoon sessions. A feature of the meeting was the discussion of the foundation of a ceramic museum in which will be displayed specimens of ceramic products of all kinds manufactured in the State of New Jersey. The nucleus of the exhibit was on display at the meeting and included a specimen of the first pottery manufactured in New Jersey. The meeting was a very satisfactory one in every respect. More than 90 were in attendance.

Adjourned.

G. H. Brown, *Secretary-Treasurer.*

ANNOUNCEMENT

1919 Summer Meeting.

AMERICAN CERAMIC SOCIETY.

Buffalo, Monday, August 4, 1919.

Members will convene in Buffalo on Monday morning, August 4th. Points of interest in and around Buffalo and Niagara Falls will be visited and the party will proceed by boat on Wednesday to Cleveland. The Buffalo local committee is endeavoring to arrange such interesting trips for the ladies as an inspection of the factory of the Larkin Company and the Shredded Wheat plant, and they will also enjoy the Falls and the ride on Lake Erie. The Iroquois Hotel has been selected as headquarters at Buffalo.

Cleveland, Thursday, August 7, 1919.

Members will arrive on the Buffalo boat at eight o'clock and will breakfast at the new Cleveland Hotel. Choice will then be given for several trips:

1. Enameled Products Company.
2. Vitreous Enameling Company.
3. Cleveland Metal Products Company.
4. Nela Park, Research "University" of the National Electric Lamp Co.
5. National Carbon Company.

There may also be an opportunity to visit the Bedford China Company, Bedford, Ohio.

The party will lunch at the Hotel Cleveland and afterwards go to the Cleveland Yacht Club where a complimentary smoker and dinner will be given by the Northern Ohio Section of the American Ceramic Society.

This post-war meeting promises to be one of the best summer meetings ever held by the Society. Members should arrange vacations so that they can combine the enjoyment of the scenic

beauties of Niagara Falls and a boat trip on Lake Erie with the social and educational features which characterize the meetings of the A. C. S.

S. C. LINBARGER,
Chairman, Buffalo Local Committee.
R. D. LANDRUM,
Chairman, Cleveland Local Committee.

Acquisition of New Members during June, 1919.

Associate.

Behrent, Leo A., Midland Terra Cotta Co., Chicago, Ill.
Blank, Wm. C., Ills. Pacific Glass Co., San Francisco, California.
Davies, J L., Clayton, Wash.
Finney, Robert S., Hotel Cumberland, New York City.
Hathaway, C. W., National Enameling and Stamping Co., Granite City, Ill.
Slater, W. M., 522 F St., N. W., Washington, D. C.
Swinnerton, B. B., 148 Taylor Ave., Beaver, Pa.
Wethey, Jr., A. H., Standard Brick & Tile Co., Portland, Oregon.

JOURNAL
OF THE
AMERICAN CERAMIC SOCIETY

A monthly journal devoted to the arts and sciences related to the silicate industries.

Vol. 2 **July, 1919** **No. 7**

EDITORIALS.

CERAMIC TERMS AND DEFINITIONS.

In the ceramic industry there has been some confusion from time to time as to the exact meaning of certain terms and in other cases it has been the practice to use several words interchangeably to indicate the same product or the same process.

Now that ceramic engineering and ceramic literature have become well established, it would seem advisable to simplify and clear up some of the terms and definitions in use.

First, there are the words "firing" and "burning" which are used interchangeably. Which is more correct, or which more desirable? Ordinarily, "burning" implies consuming by fire, reducing to ashes, injury by fire or heat (Webster). A cook may burn the toast or the cake, a child may burn its fingers, or a lamp burn a hole in a window curtain. "Burn" in all of these sentences implies either injury or destruction. It is true that Webster states that "burning" may indicate perfecting or improving by fire and heat, but this is not the popular conception of the term. In a chemical sense "burning" denotes oxidation and this again complete change of the original substance. On the other hand, to "fire" or the act of firing, means, according to Webster, to set on fire, to kindle, to ignite, or *to subject to intense heat.* It would seem, therefore, that of the primary meanings of the two words, as recognized by such authorities as Webster, the term "firing" would be more correct than "burning" as applied to the finishing of ceramic objects in the kiln. As these terms are generally or popularly understood, the term "firing" would provide a more satisfactory conception of the operation than "burning."

Of the terms "firing" and burning," therefore, the former seems more suitable and should be employed to the exclusion of the latter term.

We also desire to call attention to the words "slip" and "engobe." Webster defines the word "slip" as any covering easily slipped on (slip covers for chairs, underwaist, often of contrasting color, etc.). In this sense the word "slip" may very properly be applied to clay slips which are readily applied to surfaces of ceramic wares to provide a particular color or finish different from that of the underlying body or base. The word "engobe" is of French origin and is defined by Webster as a white or colored *slip* applied as a thin coating to earthenware, often as a support for a second glaze or enamel. If an engobe then is merely a slip, why not stick to the original term, particularly since the word slip in the ceramic sense has identically the same fundamental meaning as the term slip applied to furniture covers, wearing apparel, etc.? All evidence indicates that the term slip is entirely correct and consistent and this word should, therefore, be used in ceramic literature to the exclusion of the word "engobe."

These cases illustrate the need of adjustment in ceramic nomenclature. Other cases will no doubt come to the minds of our readers, as the frequent but incorrect confusion of the terms "fire clays" and "refractory clays," kilns and furnaces, porcelain and china, clay and kaolin, etc.

In the printing of THIS JOURNAL and in the preparation of an index for the nineteen volumes of the Transactions of the American Ceramic Society, it will be the purpose of the Committee on Publications to bring about as far as possible the elimination of superfluous or apparently incorrect expressions or terms and the coöperation of the membership of the American Ceramic Society and of the readers of this JOURNAL will be appreciated in this work.

PATRONIZING HOME MARKETS

Although the function of the American Ceramic Society is primarily to promote the arts and sciences related to the silicate industries, it would seem that the Society should be interested in fostering the continuation of those branches of the industry

which have been launched as a direct result of the war. During the past four years we have watched with pride the development and improvement of a number of ceramic products which were hitherto not manufactured in this country, or, if so, were considered of inferior quality to similar products which could be imported from abroad. We need only mention in passing; the wonderful development of our optical glass industry; the perfection and production of the highest quality of chemical glassware; the establishment of a chemical porcelain industry; the manufacture of porcelain protection tubes for pyrometers; the remarkable improvement in the quality of American spark-plug insulators; etc.

The development and production of the above wares in this country was only brought about through the painstaking researches of our investigators in our laboratories and through the expenditure of much time and money by our manufacturers in surmounting the obstacles encountered in undertaking the manufacture of new products. It is only fair that these be rewarded for their efforts. Our manufacturers do not ask that preference be given their products over products of superior quality. They are, however, entitled to consideration in competition with products of equal or inferior quality. There has been a tendency in the past on the part of the public to prefer to pay a premium for wares bearing a foreign trade mark—in spite of the fact that similar wares of equal or superior quality of domestic manufacture could be secured. This has been true to a large extent in the purchasing of ceramic wares for our industrial, college and university laboratories. This tendency has not been encouraging to our manufacturers either in the improvement of their wares or in the development of new lines.

The ceramic industry is just approaching the dawn of its development in this country. If given encouragement, our laboratories and manufacturers will over-shadow the achievements of the past four years by the progress and development which is to come. Boost for the home products!

ORIGINAL PAPERS AND DISCUSSIONS.

STRUCTURE OF GRAPHITE IN RELATIONSHIP TO CRUCIBLE MAKING.[1]

By Reinhardt Thiessen.

When the American graphite crucible manufacturers were confronted with the necessity of using American graphite, altogether or in part, as a substitute for Ceylon graphite, on which they had previously depended almost entirely, it was soon discovered that crucibles containing American graphites were very unsatisfactory, as compared with those made of Ceylon graphite.

The problem was then raised as to why crucibles made of, or containing, American graphite flakes should not be as efficient as those made of Ceylon graphite; and, if possible, to find out how to utilize American graphite flakes in crucible practice.

In this investigation it was considered desirable to make some microscopic examinations of the structures of graphite crucibles, as well as the graphite constituents themselves, and a microscopic examination, as well as a study, of the physical properties of the bonding clays used. Thirty-one samples of different makes of crucibles were examined, of both foreign and American makes.

It was soon found that the form and the structure of the graphite constituents, and the internal structure of the crucible itself, were very important factors in the successful manufacture of graphite crucibles. It is the purpose of this paper to present some of the results obtained during this investigation.

A brief discussion of the shape or form of the graphite particles will be first in order. The American graphites examined were ob-

[1] By permission of the Director, U. S. Bureau of Mines.

tained from Alabama, Pennsylvania and Canada and consist of thin plates or flakes, as they are generally called. These graphites are all similar, in that they are highly sub-laminated and may easily be reduced to still thinner flakes. The Alabama flakes are, generally speaking, the smallest and thinnest, ranging in size from that barely visible to the naked eye up to a width of about $1^1/_2$ to 2 millimeters. The Pennsylvania flake is slightly larger and thicker, and the Canadian flake averages larger than the Pennsylvania. The Madagascar flake is almost twice as large as the American flake. The size, largely due to comminution, is, nevertheless, strongly dependent on the innate structure of the graphite in question. The Alabama and Pennsylvania flakes are similar in form, as is shown in Figs. 1 and 2. There is a decided tendency towards circular and oval forms, although many irregular flakes are present in these two graphites. The Canadian flake is more irregular, although there is some tendency towards the circular and oval forms, as shown in Fig. 3.

The Madagascar flake, shown in Fig. 4, differs considerably in shape from the American graphite. Instead of a circular or oval outline, the flakes tend to be straight-edged, approaching the form of parallelograms or polygons. Many irregular forms are also present, as shown in Fig. 4. The Ceylon graphite particles are of an entirely different form, occurring in the shape of irregularly formed grains, with relatively few flake-like particles. This is shown in Fig. 5. Some of the particles are spherical and some are ovoid, while others are roughly lenticular, tetrahedral, cubical, or polyhedral with some sharp and many rounded corners and edges. Generally speaking, it may be said that the Ceylon graphite consists of irregular grains with rounded corners and edges. Cross sections of the graphite flakes and grains at the same magnifications are shown in Figs. 18 to 30.

The grain size of the Ceylon graphite, as used in making crucibles, is variable, ranging from those invisible to the naked eye to particles several millimeters in diameter or length. These sizes are largely a matter of grinding, screening and blending of the various grades thus produced, although the innate structure of the graphite particle itself exercises considerable influence.

There are, therefore, fundamental differences in the shapes and

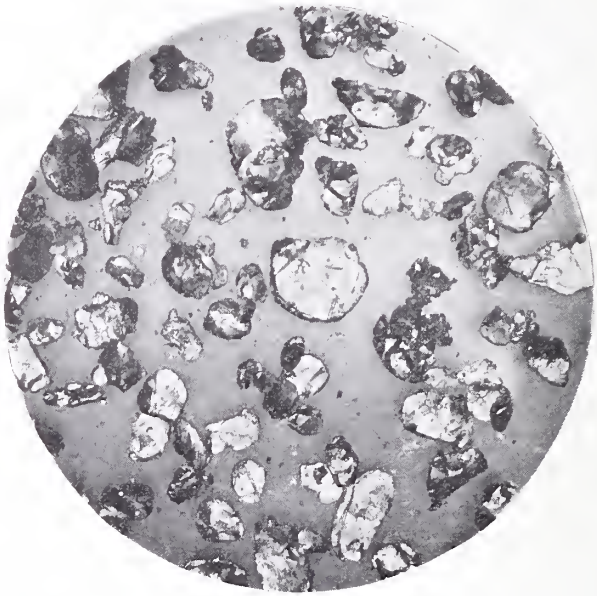


FIG. 1.—Alabama graphite flakes, seen flatwise, × 13.3.

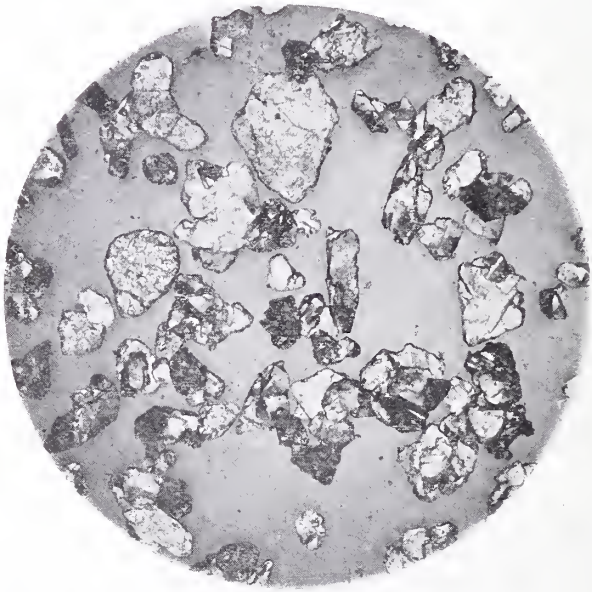


FIG. 2.—Pennsylvania graphite flakes, seen flatwise, × 13.3.

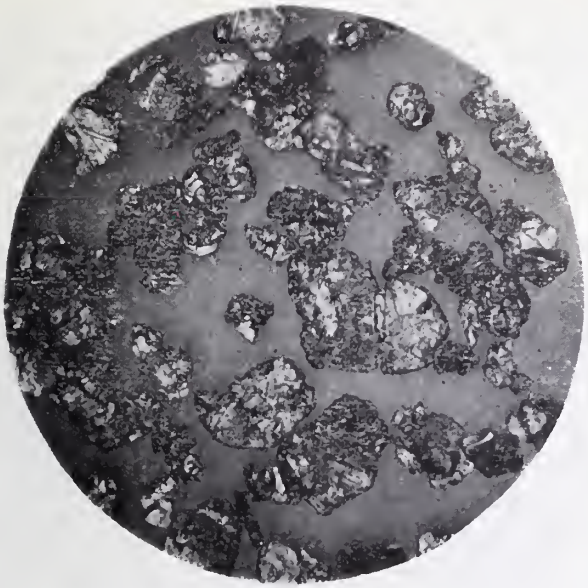


FIG. 3.—Canadian graphite flakes, seen flatwise, × 13.3.

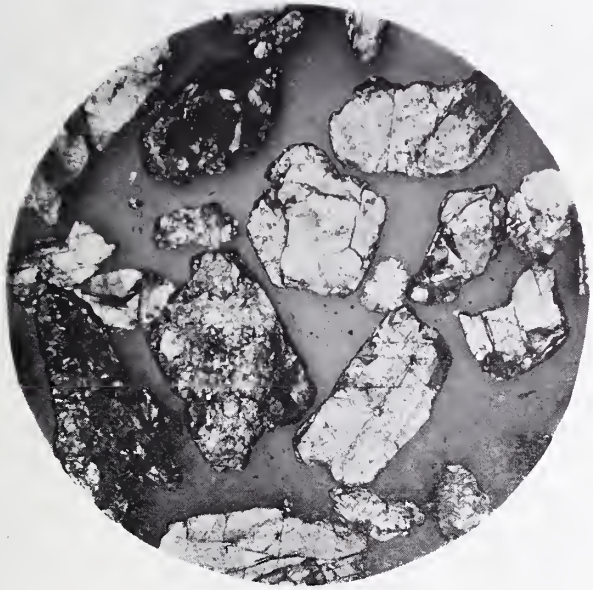


FIG. 4.—Madagascar graphite flakes, seen flatwise, × 13.3.

sizes of the American graphite flakes on the one hand, and of Ceylon graphite grains on the other hand, that naturally tend to cause great differences in their behavior under conditions of use.

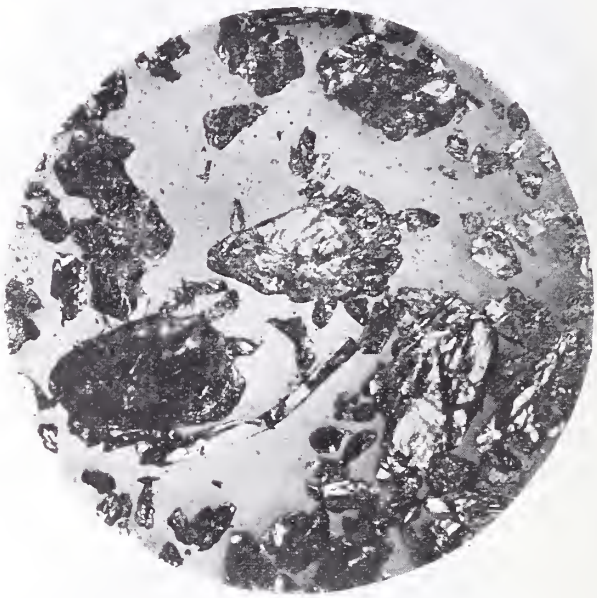


FIG. 5.—Ceylon graphite particles, × 13.3.

The Inner Structures of Graphites.

When seen in cross-section at a low magnification, the graphite constituents have a homogeneous appearance, with marks of laminations or other structures here and there. At higher magnifications, say at 200 diameters, there become visible various laminations, striations and divisions, blocking the graphite out into many forms and patterns.

All the American graphites under discussion show parallel laminations and sub-laminations into very thin sheets or flakes, the laminations being continuous along the whole length and breadth of the flake. With these graphites, the laminations are most pronounced in the Canadian flake and least pronounced in the Alabama flake. The Madagascar flake shows similar and

more definite laminations than the Canadian. See Fig. 6 (Canadian), Fig. 7 (Alabama), and Fig. 8 (Madagascar), all at 133 diameters. It will be noticed that the Alabama flake has rather rough surfaces, as seen in cross-section, compared with either the Pennsylvania, Canadian or Madagascar flake, giving, in general, a more irregular cross-section.

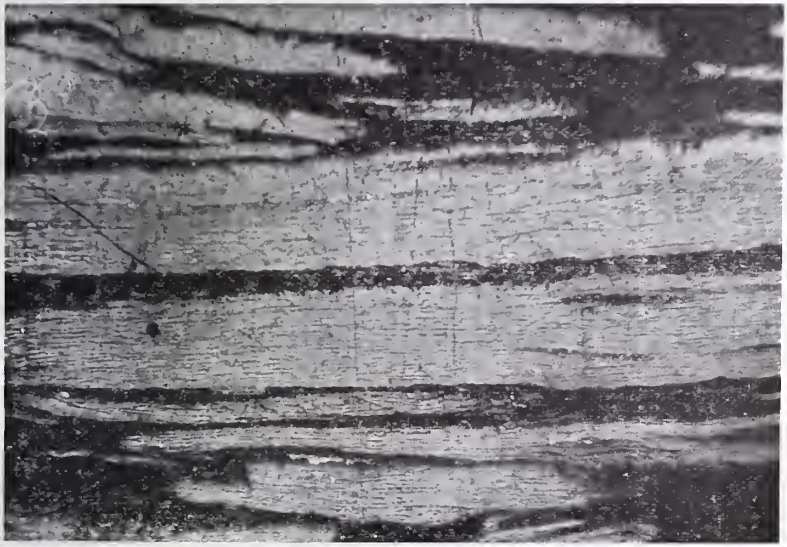


FIG. 6.—Cross section of Canadian graphite flakes embedded in clay, at a magnification of 133 diameters.

The Ceylon graphite has an entirely different structure, which may be subdivided into two main types. In the one (the least prevalent type) there is a parallel arrangement of very thin laminae. These are, however, not continuous from side to side of the grain, but are blocked off into more or less regular areas, when seen in cross-section, by means of breaks at right angles to the laminae (see Fig. 9, 133 diameters). The laminations are not necessarily parallel to the outer surfaces, as in the American flakes; in fact, they usually run diagonally from one surface to the other. Hence, the mass or grain is more or less regularly blocked off into forms approaching the parallelopiped and cube.

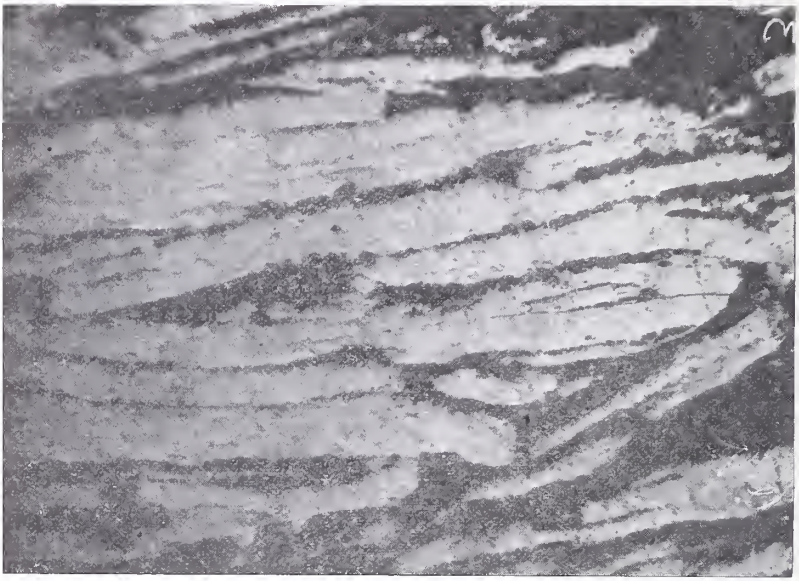


FIG. 7.—Cross section of Alabama graphite flakes embedded in clay, at a magnification of 133 diameters.

FIG. 8.—Cross section of Madagascar graphite flakes embedded in clay, at a magnification of 133 diameters.

FIG. 9.—Cross section of Ceylon graphite grains embedded in clay, at a magnification of 133 diameters.

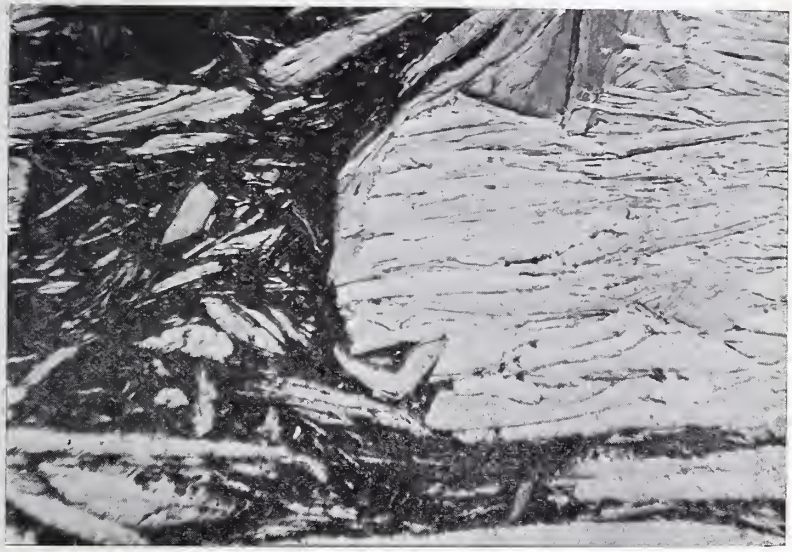


FIG. 10.—Cross section of Ceylon graphite grains embedded in clay, at a magnification of 133 diameters.

These sub-divisions are further composed of very thin plates, which are discussed later. In the other type of Ceylon graphite, which occurs more frequently, the grains are sub-divided into more or less regular triangular prisms and parallelopipeds. Figs. 10, 11 and 12 (133 diameters) represent this type. These prisms or blocks are further composed of very thin plates, parallel to each other, but variously arranged with respect to those of other prisms or blocks, as shown in the illustrations. A large variety of patterns are thus formed, due both to the different positions of the group of thin plates forming the prisms or blocks and the shapes of the prisms or blocks themselves.

This structure is vividly illustrated by the structure of the so-called graphitic acid. When Ceylon graphite is subjected to Brodie's reaction (oxidation with potassium chlorate and nitric acid), a yellow substance, called graphitic acid or graphitic oxide, is formed. The structure of this product is interesting. Even at the low magnification of 66 diameters (Fig. 13), it is shown to consist of groups of numerous thin plates closely packed and of a relatively few amorphous-appearing granules. When these are submersed in a dilute solution of ammonia, the whole mass apparently swells up, due to a slight separation of the thin plates from one another (Fig. 14). The mass thus loosened up is in a favorable condition for observation.

It is now clearly shown that the graphitic oxide "crystals" consist of the blocks or prisms, or parts of the same, that originally formed the graphite grains. The ultimate plates which formed such aggregates have been impregnated, partially separated, and have become translucent, or transparent, through the reaction. Under the ultra-microscope these plates are shown to be composed of ultra-microscopic particles or micellae closely cemented together in the same manner as clay, except that the clay particles are very much larger. They must, therefore, be considered colloidal rather than crystalline.

The grains or particles of the Ceylon graphite are, therefore, very complex objects. No matter into how small sub-divisions the grains are broken, nor in what direction they break, the fracture is apt to be an irregular grain and rarely a flake.

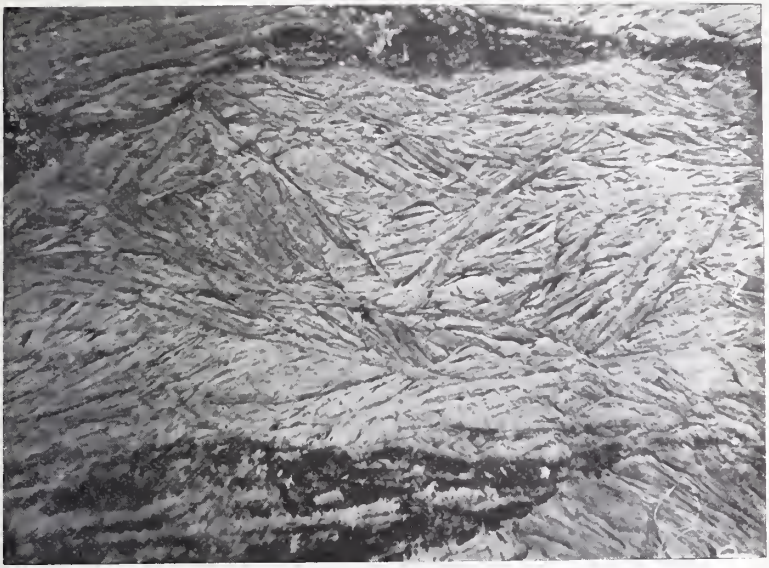


FIG. 11.—Cross section of Ceylon graphite grains embedded in clay, magnified 133 diameters.

FIG. 12.—Cross section of Ceylon graphite grains embedded in clay, magnified 133 diameters.

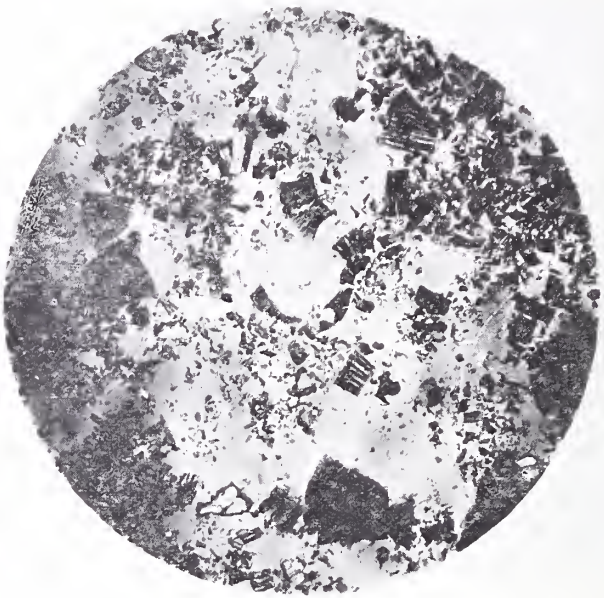


FIG. 13.—"Graphite oxide" floated out in ammoniacal water. Many groups of thin plates are distinguishable, × 66.

Structures Observed at High Magnifications.

When sections of graphite are cut at right angles to the laminae and observed at a magnification of from 1000 to 2500 diameters, the laminae shown at a magnification of 133 diameters (Figs. 9 to 12) are found to be still further sub-divided into sub-laminae. Fig. 15 (666 diameters) shows this in a Canadian graphite flake. It will be noticed (Fig. 15) that the photograph shows a number of bands quite definitely marked off, and that each of these bands is further sub-divided into a considerable number of less definitely marked bands. Each of the faintly marked bands represents a thin plate. When graphitic acid is formed from graphite, a more or less complete separation of the plates is effected. Since the magnification of Fig. 15 is 666 diameters, direct measurement will readily show that the more definitely marked plates are from 5 to 10 microns thick, while the less definitely marked plates are in the neighborhood of only 1 micron in thickness

All the graphites examined reveal a similar layering under high magnification, provided the sections are cut at right angles to the laminae. In view of the very many positions, a compilation of such plates may assume in the Ceylon graphite, sections of the prisms in every position from cross-sections to parallel sections are met with in this variety, as shown in Figs. 10 and 12.

FIG. 14.—Two groups of graphitic oxide plates at a higher magnification. In the one the plates are seen edgewise and in the other flatwise, × 66.

Very often structures are revealed that have somewhat the appearance of crystalline particles or granules. The definiteness of the graphite structure, the shape and size of the plates, the compactness of the mass, the close or loose packing of the particles, all vary considerably in the different graphites examined, as well as in the different flakes or grains of the same graphite, although each kind possesses characteristic qualities.

FIG. 15.—Cross section of a Canadian graphite flake at a high magnification, × 666.

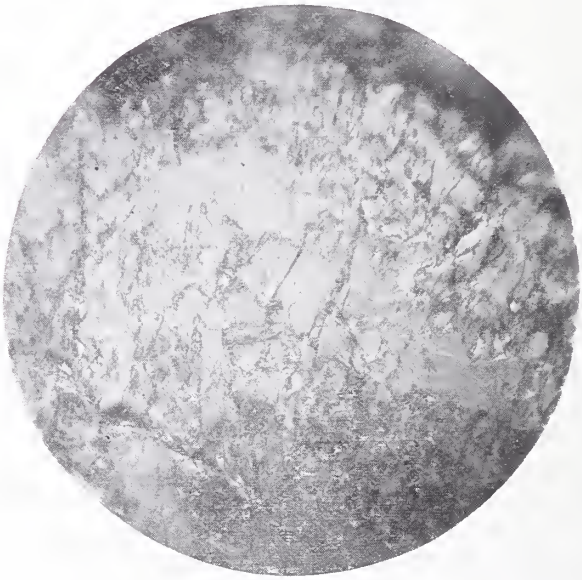


FIG. 16.—Cross section of an Alabama graphite flake at a high magnification, × 666.

Fig. 16 (x 666) shows a section of the Alabama flake and represents an appearance frequently found. It will be seen that the mass, which appears amorphous at a magnification of 200 diameters, is here resolved into very minute particles of a crystalline appearance. Fig. 17 (x 666) represents a section of the Madagascar flake. It is noticeable that the crystalloid particles are here considerably smaller than in the Alabama flake. In fact, in Fig. 17, they are so small that they are just within the limit of visibility. In other cross-sections, however, these particles approach those of the Alabama flakes in size. The arrangement, size, form and proximity to one another of the particles in the Ceylon graphite vary considerably, but, in a general way, are very similar to those of the other graphites.

The question now arises whether or not these particles are really crystalline. Many of them appear completely homogeneous at any magnification that they were subjected to. Very many, on the other hand, are resolved into ultra-particles or micellæ, and appear non-crystalline. It is very probable that all the particles resolve into ultra-particles under proper illumination and lenses of high resolving power. This supposition is borne out by the ultra-structure of graphitic acid.

The Arrangement of the Flakes in the Crucible.

An English crucible, in which all the graphite is Madagascar flake, has been pointed out to be one of the most durable pots on the market. The structure of the body of this make of crucible will be taken up first and will serve as a standard of comparison. A cross section of this crucible is shown in Fig. 18 at a magnification of 13.3 diameters. The white linear patches represent the cross sections of the graphite flakes; the darker rounded or irregular areas represent the sand particles present in most graphite crucibles as one of the ingredients, while the grayish mass between these two constituents represents the clay binder and the clay mixed with finer particles of graphite.

It will be noticed that the flakes are arranged, in general, parallel to each other, and approximately parallel to a line tangent to the outer surface of the crucible-wall, in quite uniform and regular order. In a measure, the flakes overlap each other, shingle

FIG. 17.—Cross section of a Madagascar graphite flake at a high magnification, × 666.

FIG. 18.—Cross section of a portion of a crucible wall in which the graphite is all Madagascar flake, × 13.3.

fashion. Only occasionally is this order departed from. Occasionally, also, some of the flakes are grouped more or less, but this grouping is slight. The flakes themselves are of a rather uniform shape and of uniform sizes, grading gradually from the largest to the smallest.

It is noticeable, also, that the flakes are closely packed together. This feature is well shown in Fig. 8, representing cross sections, at a magnification of 133 diameters, of the medium to the smaller flakes. The uniformly parallel arrangement in general, and the regular plate-like shape of the flakes are the prominent features in the structure of this crucible.

The sand in this crucible is shown to be irregularly distributed. Fig. 18 reveals but a few particles of sand, which are rather variable in size and sharp cornered.

A sample of an American make of crucible, containing Canadian flakes only, is shown to be similar in structure and in the arrangement of the flakes to the English make. The uniformity in arrangement and the parallel layering of the graphite flakes appears to be even better than that in the English. The shingle-like overlapping is quite apparent in Fig. 19.

While the flakes of the Canadian graphite are similar to the Madagascar flakes in form, it will be noticed that a certain number of them are slightly lenticular. The flakes in this sample are, however, not nearly as closely packed as those in the English make of crucible. This is clearly shown in a comparison of Figs. 18 and 19. This observation is in harmony with the fact that the percentage of graphite, *viz.*, 40 per cent, is rather low. The sand particles in this sample are comparatively small, but well distributed through the body.

The structure of a Japanese crucible, the source of the graphite flakes of which has not been ascertained, is also similar to that of the English make of crucible. Although the general shape of the flakes is similar to those of the two foregoing samples, they are not quite of the same uniformity of shape; also, the range of sizes is greater, a considerable number of the flakes being both broader and thicker, but the plate-like shape is retained in general. The sand particles are large and rounded and well distributed through the body. The flakes appear to be quite closely packed, closer than

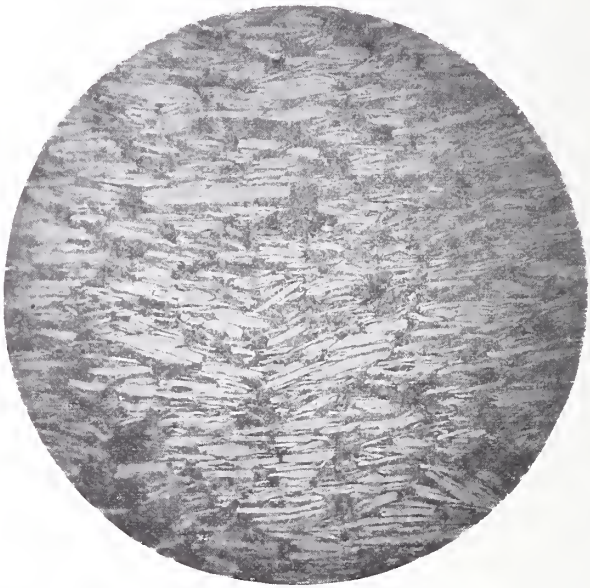


FIG. 19.—Cross section of a portion of a crucible wall in which the graphite is all Canadian flake, × 13.3.

FIG. 20.—Cross section of a portion of a Japanese crucible wall, graphite unknown, × 13.3.

the sample containing Canadian graphite, but not as close as in the English sample (see Fig. 18).

The cross section of the wall of a crucible, containing Ceylon graphite only, presents an entirely different appearance. In this, there is not nearly that systematic and uniform arrangement of the graphite constituents as has heretofore been the rule. In fact, great irregularity is noticed. In some crucibles the flakes are jumbled together in a chaotic condition, while in others there is a fairly good systematic arrangement. These conditions are fairly well represented in Figs. 21, 22 and 23, and represent about all the varieties of conditions of all the pots containing all Ceylon graphite. Fig. 21 shows hardly any orientation of the graphite constituents; the angularity, irregularity, the isodiametric qualities, and the great range in sizes of the particles, stand out prominently, and are at once noticeable. In Fig. 22, there appears a considerable degree of orientation. Notice that the particles are, on the whole, more plate-like than those shown in Fig. 21. The great range in sizes is also particularly noticeable here, a very large proportion of the graphite being in the form of fine dust. The condition shown in Fig. 23 differs in that it contains a relatively large proportion of large particles and a relatively small amount of the finer matter.

Fig. 24 represents a section of a crucible in which there is 55 per cent graphite, 25 per cent of which is said to be Canadian flake. The Canadian flake may readily be distinguished from the Ceylon through the difference in shape.

Figs. 25 and 26 represent sections of crucibles in which 90 per cent of the graphite is Ceylon and 10 per cent is Pennsylvania. The Pennsylvania flake here again is easily distinguishable from the Ceylon graphite. The bodies of these crucibles were of poor consistency and brittle, giving rise to unfinished surfaces, as may be seen in the figures.

Fig. 27 represents a section of a crucible reported to contain 51 per cent graphite, 75 per cent of which was said to be Ceylon and 25 per cent to be domestic flake.

Fig. 28 shows a similar appearance; yet the nature and arrangement of the flakes, which are evidently Alabama, differ.

Fig. 29 represents a cross section of a crucible containing 52.5

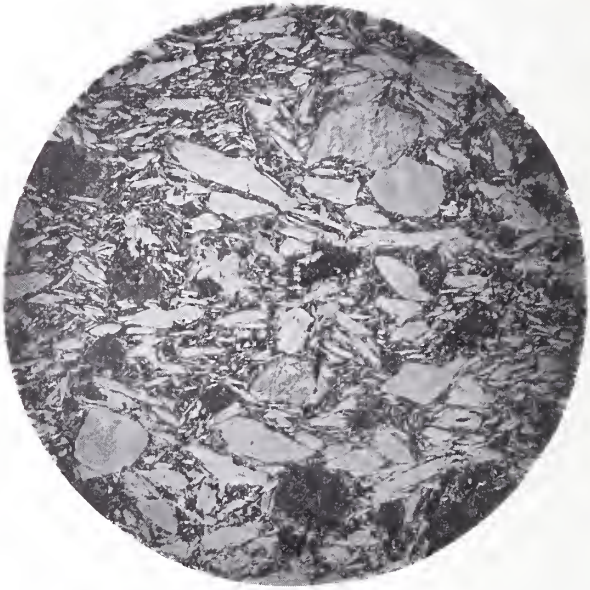


FIG. 21.—Cross section of a crucible wall in which the graphite is all Ceylon, × 13.3.

FIG. 22.—Cross section of a portion of a crucible wall in which the graphite is all Ceylon, × 13.3.

FIG. 23.—Cross section of a portion of a crucible wall in which the graphite is all Ceylon, × 13.3.

FIG. 24.—Cross section of a portion of a crucible wall containing a total of 55% graphite, 75% of which is Ceylon and 25% Canadian flake, × 13.3.

FIG. 25.—Cross section of a portion of a crucible wall in which 90% of the total graphite is Ceylon and 10% Pennsylvania flake, × 13.3.

FIG. 26.—Cross section of a portion of a crucible wall in which 90% of the total graphite is Ceylon and 10% Pennsylvania flake, × 13.3.

FIG. 27.—Cross section of a portion of a crucible wall said to contain a total of 51% graphite, 75% of which is Ceylon and 25% domestic flake, × 13.3

FIG. 28.—Cross section of a portion of a crucible wall, in which 83.5% of the total graphite is reported to be Ceylon and 17.5% domestic flake, × 13.3.

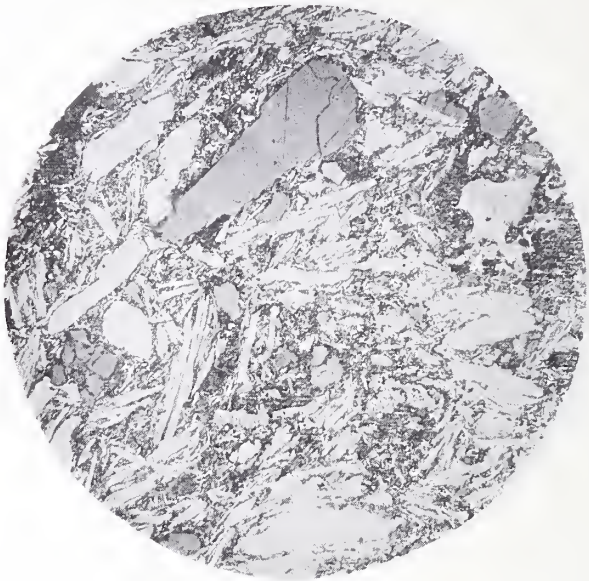


FIG. 29.—Cross section of a portion of a crucible wall containing 52½% graphite, 85% of which is Ceylon and 15% Madagascar flake, × 13.3.

FIG. 30.—Cross section of a portion of a crucible wall in which 65% of the total graphite is Ceylon, 20% Alabama flake and 15% Madagascar flake, × 13.3.

per cent graphite, 85 per cent of which is Ceylon and 15 per cent of which is Madagascar flake. The large differences in the sizes of the Madagascar flakes shown in the figure, over that of the Alabama and the Pennsylvania flakes, shown in the previous figures, is clearly evident.

Fig. 30 represents a section of a crucible in which the graphite consists of 65 per cent Ceylon, 20 per cent Alabama flake, and 15 per cent Madagascar. Here also the three kinds of graphite constituents are distinguishable.

The Effect of Graphite Characteristics on the Graphite Crucible.

Having analyzed the relative sizes, forms and structures of the graphite constituents and the structure of the crucible itself, it now remains to correlate the findings with the essential question of the crucible problem, as stated at the outset. Unfortunately, not enough data are at hand to give a definite answer. This part of the investigation, as in all other phases relative to this problem, should be looked upon as an entering wedge towards the solution of a most important commercial problem. It is hoped that all crucible manufacturers will use these data and study their ware in a similar way, and connect and correlate the data with an aim towards the improvement of their product and finally make the data and findings available for the trade in general.

A few suggestions and conclusions of the findings, nevertheless, may be briefly outlined.

In looking over the cross sections of the walls of different crucibles, various factors that entered into the structure of these became apparent. The factors follow:

(*a*) The arrangement of the flakes.

(*b*) The form of the flakes.

(*c*) The different sizes of the flakes; in other words, whether fine or coarse, as a whole.

(*d*) The large range of sizes.

(*e*) The proximity of the graphite constituents.

(*f*) The total surfaces of the constituents in relationship to their volumes.[1]

(*g*) The physical conditions of the clay binder and the sand and other ingredients that are invariably found in the make-up of the crucible.

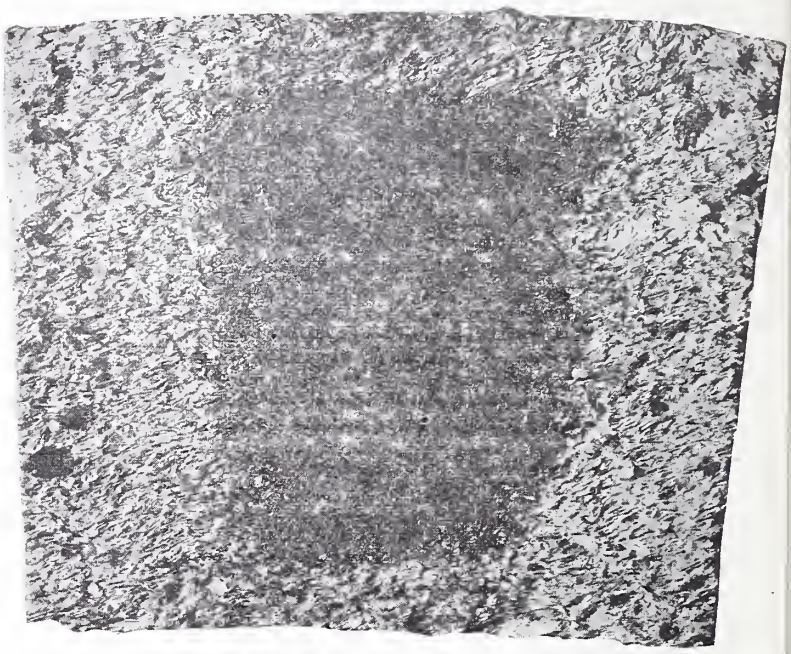


FIG. 31.—Cross section of a block sawed from the wall of an English crucible containing Madagascar graphite flake only, a cross section of which is shown in Fig. 18, subjected to a temperature of approximately 1250° C. in a muffle furnace, for $7^1/_2$ hours. (× 3.)

An important factor in the efficiency of a crucible appears to be the arrangement of its graphite. Theoretically, everything else being equal, a pot in which the graphite is in the form of flakes arranged parallel to each other and parallel to a tan-

[1] Bleininger, A. V., "Recent Developments in Ceramics," *J. Ind. Eng. Chem.*, **10**, 844–849 (1918). Bleininger, A. V., "Notes on the Crucible Situation," *J. Am. Inst. Metals*, **11**.

gent to the outer wall of the pot, should be the most resistant to oxidation under the conditions to which the crucible is subjected during use. A few experiments will readily verify this theoretical conclusion. Cubical blocks were sawed out of various makes of crucibles and all were subjected, as far as possible, to the same conditions in a muffle furnace heated to a temperature of approxiamtely 1250° C for $7^1/_2$ hours. In every case, excluding other factors, it was found that oxidation was least on the sides parallel to the flakes or flat-sides of the graphite grains and most on the sides perpendicular to the flakes or longer axes of the graphite constituents. The English make of crucible, containing all Madagascar flakes (a cross section of which is shown in Fig. 18), shows these results most emphatically. Fig. 31 shows the interior of a block cut out of the same crucible and exposed to the aforesaid conditions. The outer, light zone represents the burned-out part, while the dark inner area represents the unburned portion. It will be noticed that the graphite has burned out the least on the sides of the block that formed the inner and the outer surface of the crucible wall; that is, on the sides parallel to the flakes, while on the sides that were sawed out of the wall, the sides perpendicular to the flakes, the graphite has burned out to a depth almost three times as great.

The same phenomenon, although not nearly as pronounced, is shown in the crucible containing all Canadian flake, a cross section of which is shown in Fig. 19. Fig. 32 shows a cross section of a block of this crucible subjected to exactly the same conditions as the other. Here, also, the graphite has been burned out the least on the sides that formed the outer and the inner surfaces of the wall of the pot, or the sides parallel to the largest surfaces of the flakes, while it is burned out to the greatest depth on the sides perpendicular to the flakes. It will be noticed, however, that in this case the graphite had not been burned out nearly as deep as in the previous case (the English crucible). Another clear-cut example is shown in a crucible containing 83.5 per cent Ceylon graphite and 17.5 per cent Alabama flake, shown in cross section in Fig. 28. Fig. 33 represents the interior section of a block of this crucible subjected to the same conditions as the others; and again, the sides parallel to the flakes have oxid-

ized the least and the ones perpendicular to the longer axes, the most, but very much deeper, on either side, than any of the specimens heretofore shown.

If now a hypothetical case is taken in which all the graphite

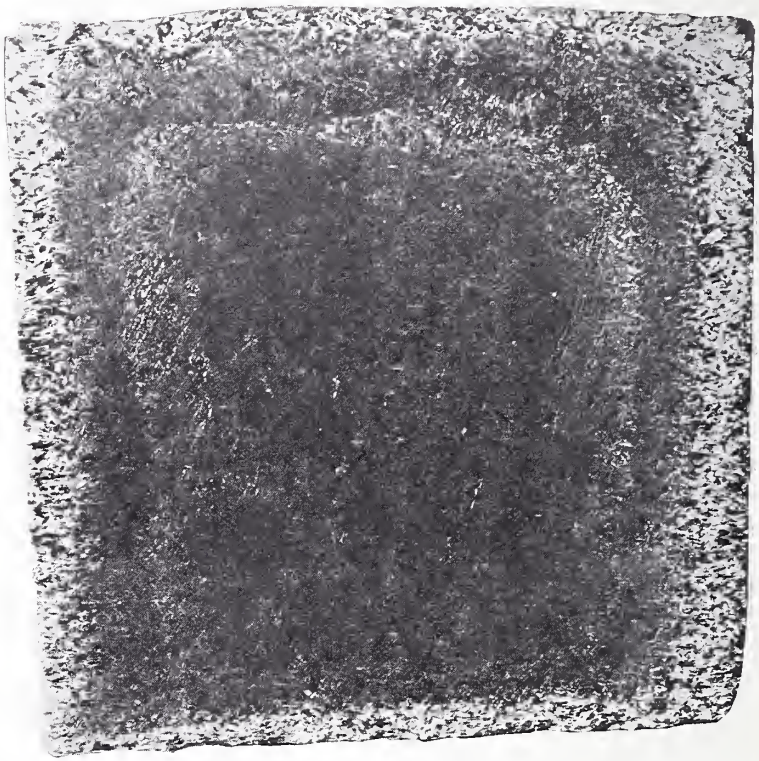


FIG. 32.—Cross section of a block sawed out of a crucible wall containing Canadian flake only, shown in cross section in Fig. 19, and subjected to a temperature of approximately 1250° C. for $7^1/_2$ hours. (× 3.)

flakes, any Canadian flakes, were arranged perpendicular to the tangent to the outer wall of the crucible, all other factors being disregarded, the whole wall of the pot would be oxidized through and through under a few heats under similar conditions to which these sample blocks were subjected; or, if the crucible contained

only Ceylon graphite consisting of relatively large grains of approximately uniform diameter, the same thing would happen.

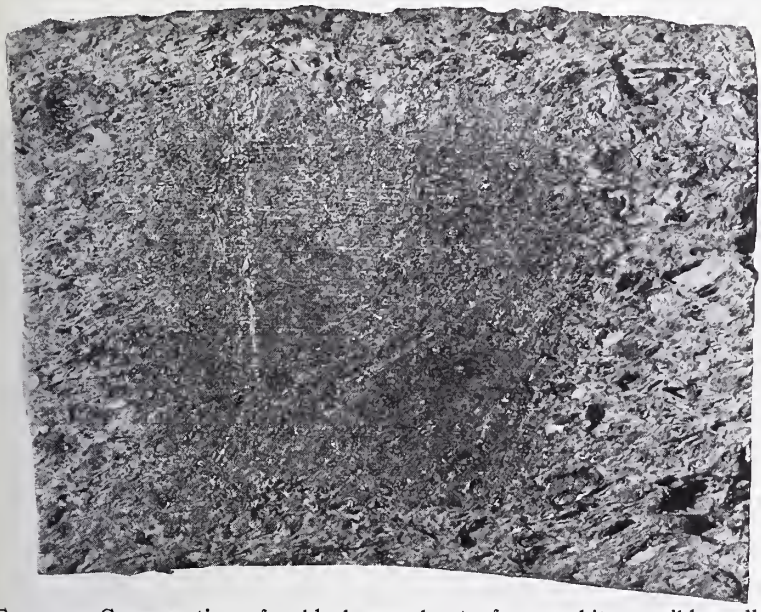


FIG. 33.—Cross section of a block sawed out of a graphite crucible wall, shown in cross section in Fig. 28, and subjected to a temperature of approximately 1250° C. for 7 1/2 hours. (× 3.)

Other Factors Involved.

The great differences in the depths to which oxidation of the graphite has penetrated leads to the conclusion that other factors are involved. One such factor is easily discovered in the fineness or coarseness of the constituents, a factor which connects up directly with the foregoing. This is shown in a comparison of the examples already given. The specimen containing the Canadian flake (Figs. 19 and 32), in which the texture is relatively fine, should be compared with the English specimen containing all Madagascar flake (Figs. 18 and 31) and the one shown in Figs. 28 and 33, in which the textures are considerably coarser. The relatively greater depth of oxidation is clearly seen.

To go one step further in this direction, Figs. 34 and 35 are

offered for examination, both from pots in which the graphite consists of 90 per cent Ceylon and 10 per cent Pennsylvania flake and shown in cross sections in Figs. 25 and 26. This material is still coarser than any previously shown. In Fig. 34, the graphite

FIG. 34.—Cross section of a block sawed out of a crucible wall containing 90% Ceylon and 10% Pennsylvania flake, also shown in cross section in Fig. 25, and subjected to a temperature of approximately 1250° C in a muffle furnace for $7^1/_2$ hours. (× 3.)

is seen to be almost completely burned out, only a small core being left in the center. In the one shown in Fig. 35, the coarsest

of them all, the graphite is burned out completely. In Fig. 34, the rule of burning out most on the side perpendicular to the longer axis is followed quite closely.

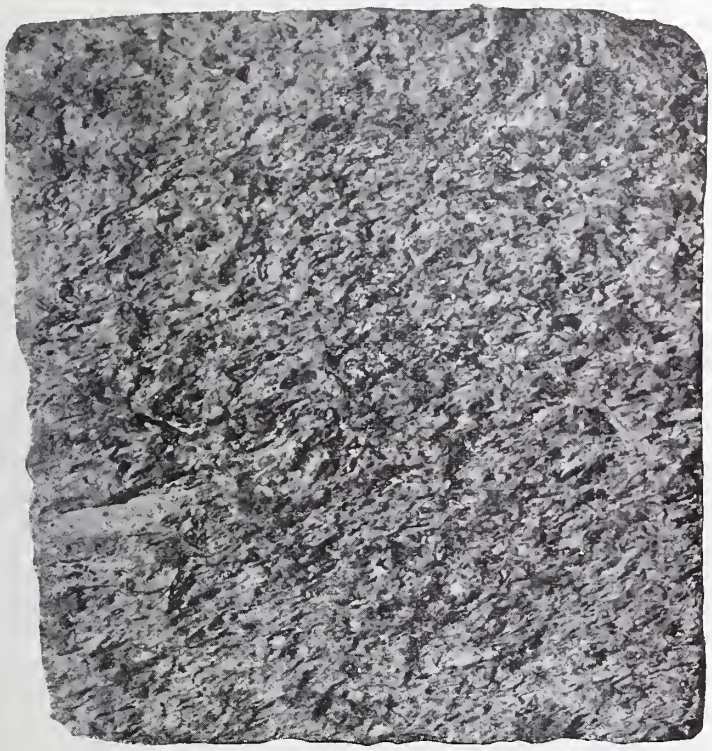


FIG. 35.—Cross section of a block sawed out of a crucible wall in which the total graphite consists of 90% Ceylon and 10% Pennsylvania flake, also shown in cross section in Fig. 25, and subjected to a temperature of approximately 1250° C in a muffle furnace for 7½ hours. (× 3.)

It is safe to say that the sizes of the graphite constituents, especially the graduation of sizes (that is, whether the particles are, on the whole, coarse, medium or fine and whether there is a uniform graduation of sizes from the smallest to the largest) play an important part in the resistance to oxidation; but there are other

factors involved that must first be eliminated and their influence determined, each for itself, before any definite statements can be made.

The Plasticity of the Clay Used as a Bond.

The plasticity of the clay is of the utmost importance, and upon this the plasticity of the graphite and clay mixture must largely depend. But the clay by no means determines this alone, since the plasticity depends also upon the form of the graphite constituents used.

In a mixture of clay and graphite particles, in which the latter are in the form of grains of a more or less uniform diameter, and in which the sizes graduate more or less uniformly, the plasticity of the clay, while it is somewhat reduced, is reduced to a minimum degree. The mixture, under pressure, will flow in any direction without rupturing. In a mixture in which the graphite is in the form of flakes or thin plates, the plasticity will naturally be reduced to a far greater degree, because the mass is not equally free to flow in all directions under pressure, being interfered with to a larger degree in the direction perpendicular to the plane of the flakes than in any other, and being least checked in the direction parallel to the flakes. The flow will naturally tend to go in this latter direction. Any movement in the direction perpendicular to the flow of the flakes will result in a disarrangement of any parallel layering, and tend to shear and rupture the bond, while a movement in the direction parallel to the flakes will tend to cause too great slipping while the pot is being molded. A mixture with Ceylon graphite (grains instead of flakes) will, for these reasons, be more uniformly plastic, more easily brought into the proper form and with less danger of rupturing than a mixture containing graphite flakes.

Peptization of Clays.

Clays as used in crucible making must first be very finely ground. But no matter how finely ground they may be they are mostly in the form of particles. Under the microscope most of these appear as coarse particles. Clays in this form, after being mixed and kneaded with water, are still largely in the form of

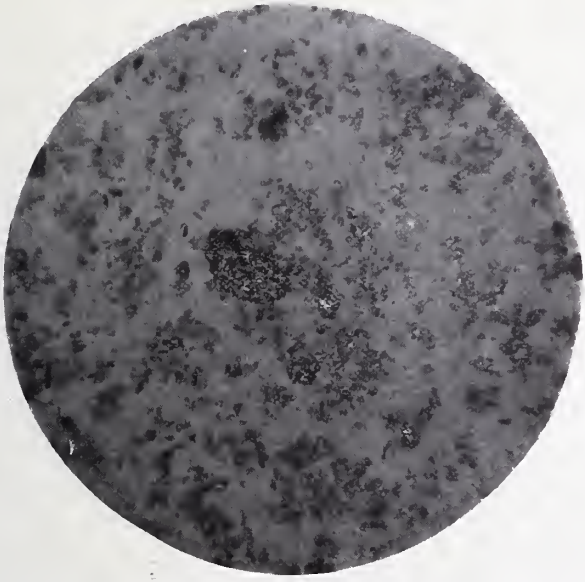


FIG. 36.—Unpeptized clay, graphite and water mixture, from which the graphite has largely been removed. (× 166)

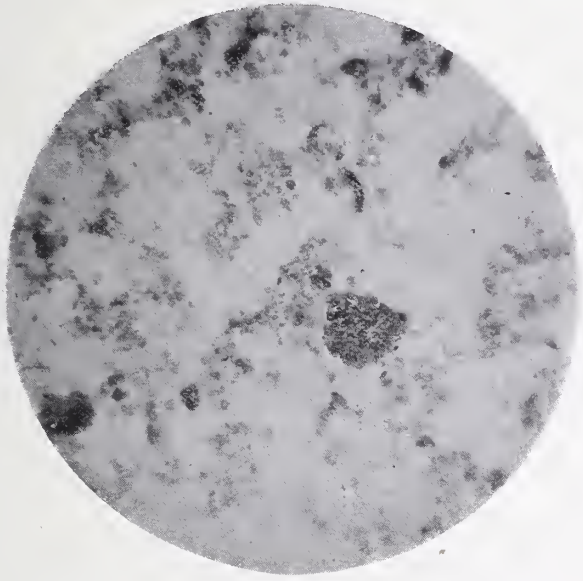


FIG. 37.—Unpeptized clay, mixed and kneaded with water. (× 133.)

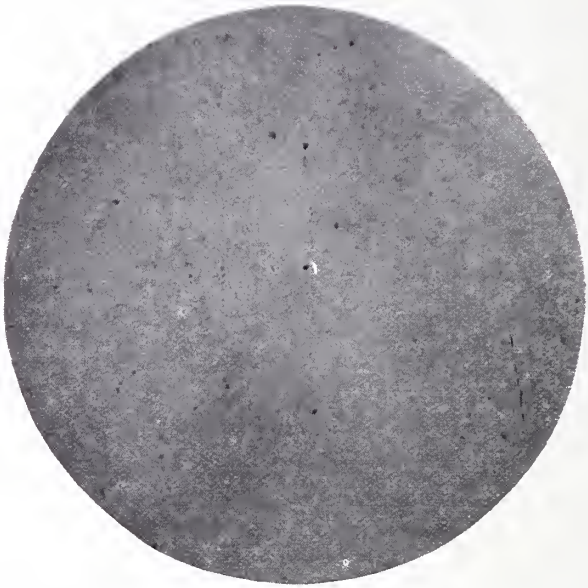


FIG. 38.—Peptized clay, graphite and water mixture from which the graphite has been largely removed. (× 133.)

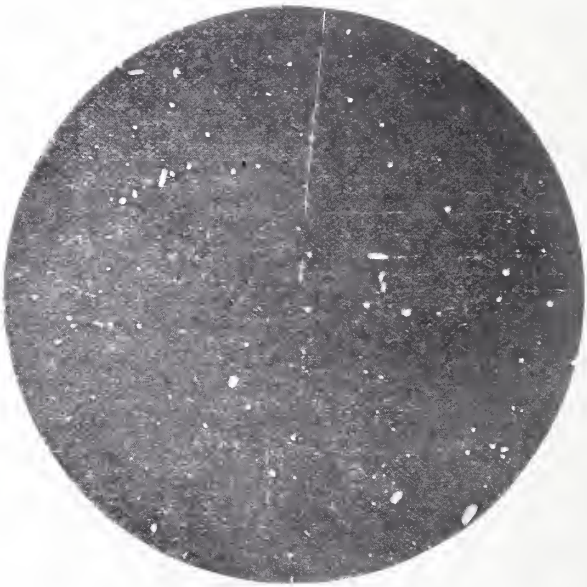


FIG. 39.—The ultra clay particles from the mixture, shown in Fig. 38. (× 1666.)

coarse particles and are only partially reduced to their ultra-particles.

It is possible, however, to disperse clays completely by means of proper reagents; in other words, they may be separated into their ultra-particles, or peptized, as shown in Figs. 38 and 39.

The specific surface of clay, when thoroughly dispersed, has been increased enormously over that of the unpeptized clay or clay in only a partially dispersed condition. The specific surface in the graphites is an important factor in graphite-crucible efficiency. The more plate-like and the thinner the flakes the greater is the specific surface. Hence, the total surface of the Alabama flake graphite over that of the Ceylon graphite must be very large. However, this greater surface can be more than counter-balanced by thoroughly dispersing the clay. A closer proximity and a more tenaceous adherence of the graphite flakes to one another is then made possible and, at the same time, the flakes are free to move or slip over one another due to the lubricating action of the film of finely-divided clay particles covering each graphite flake.

Fig. 37 shows the original clay (Dorset) mixed and kneaded with water. Fig. 36 shows an unpeptized clay and graphite mixture, from which the graphite has largely been removed. Fig. 38 shows a mixture of peptized clay and graphite, the graphite having largely been removed. Fig. 39 shows the ultra-particles of the clay at a very high magnification.

Summary.

Many crucibles containing Ceylon graphite were examined; also, an English crucible containing Madagascar graphite, several American crucibles containing American graphite, and one Japanese crucible (graphite origin unknown) were examined.

The American graphites (Canadian, Alabama, and Pennsylvania) and the Madagascar graphite have a flake formation, while Ceylon graphite has a more granular form.

Some factors entering into the structure of graphite crucibles follow:

(*a*) If the graphite is in the flake form, the flakes should be parallel to each other and parallel to the tangent to the outer

wall of the crucible. This arrangement tends to minimize oxidation. More care must be used in making crucibles containing flake graphite, as the clay-graphite mixture will not flow in all directions, due to the unsymmetrical shape of the admixed graphite particles.

(*b*) It is better to have the graphite particles uniformly small, rather than uniformly large. If mixed sizes are present, it is advantageous to have an even graduation from the largest to the smallest.

(*c*) The graphite particles should be uniformly and closely packed together.

(*d*) The total surface of all constituents, in relationship to their volume, is a factor that has long been recognized.

(*e*) The clay should be in a suitable physical condition to make it plastic even after the addition of the graphite.

Acknowledgments.

The writer is deeply indebted to Mr. F. L. Arnsberg and Mr. Arthur J. Jackman of the Vesuvius Crucible Company, for their willing and enthusiastic coöperation, and for imparting their valuable experience, thus being of great help in the work. The Vesuvius Crucible Company and Mr. George D. Dub also rendered valuable assistance in furnishing much of the material required.

The writer wishes further to express his gratitude to Mr. D. A. Lyon, Supervisor of Stations, and Mr. A. C. Fieldner, Supervising Chemist, who did much to facilitate the work; and also to Mr. R. T. Stull, Superintendent of the Ceramic Station of the Bureau of Mines at Columbus, Ohio.

Chemical Division,
Pittsburgh Experiment Station,
Bureau of Mines,
Pittsburgh, Pa.,
May 9, 1919.

THE COOLING OF OPTICAL GLASS MELTS.

By Howard S. Roberts.

When a melt of glass is removed from the melting furnace, it may be either left to cool in the pot, or be cast and rolled into a plate. This paper has to do with the former case, where the melt is cooled in the pot.

The choice between these two methods is a question of the quality of glass desired and of expediency. When the melt is set out of the furnace it is never entirely homogeneous. The middle is usually free from contamination, but near the bottom and sides of the pot, the glass is badly contaminated with material dissolved from the pot. If the melt is cooled in the pot, this contaminated glass need not be much disturbed, but where it is cast and rolled into a sheet, it is apt to be spread rather evenly through the whole plate.

While the process of cooling in the pot involves a greater amount of labor and waste in the mechanical preparation of the glass, it can be made to yield glass of the highest quality. The rolling process, on the other hand, may give a large yield of glass that is entirely suitable for many, if not most, optical instruments.

Where the melt is cooled in the pot, the conditions to be sought are: (1) that the cooled melt shall break into large, reasonably rectangular blocks, having smooth faces; and (2) that not much glass shall be spoiled by inhomogeneity, *i. e.*, by striae, bubbles, crystals or devitrification.

It is not feasible to cool the melt so slowly that it will remain unbroken; nor is this particularly desirable because of the necessity of breaking it up later for examination and for rejection of defective portions.

At the melting temperature, glass is a fairly mobile liquid. As it cools its viscosity increases continuously, until at ordinary temperatures, though probably not infinite, the viscosity is so great that we apparently have an elastic solid. For convenience

we shall divide the whole range of cooling into three stages: The Stage of Fluidity; The "Annealing Range;" The Final Stage.

Stage of Fluidity.

During this stage, the glass is apt to be spoiled by striae due to convection; by the formation of vacuum bubbles, and, in certain glasses, by the formation of one or more crystalline phases in the glass itself.

Convection.—On leaving the furnace, the glass is still quite fluid, having been stirred until the time of setting out. The portion close to the sides and bottom of the pot is richer in material dissolved from the pot and has an index of refraction different from that of the glass nearer the center. As the melt cools convection currents tend to draw streamers of this contaminated glass upward through the middle of the melt. Below the "setting-out temperature" the glass is too viscous to readily assimilate these streamers by solution; so that, having once appeared, they remain as "striae" or "ream." Particles of foreign material, such as bits of the pot, may also be carried to the middle of the melt where they usually float upward or settle, dragging tails of contaminated glass behind them. Convection continues at a decreasing rate as viscosity increases on cooling, and soon becomes inconsiderable.

As convection is caused by the settling of denser, cooler glass, it can be reduced by insulating the upper portion of the melt, and cooling rapidly from the bottom. Unfortunately, present melting practice usually insures that the bottom of the pot, on leaving the furnace, shall be at a somewhat higher temperature than any other portion; moreover, the bottom is usually made much thicker than the sides. Thus it has not been found possible entirely to prevent convection even by immersing the bottom of the pot in a pan of water.

It should be emphasized that measures to reduce convection should be taken immediately on setting the pot out of the furnace. The surface of the melt should immediately be covered with a layer of kieselguhr or other light insulator; and the pot be placed on bricks as quickly and with as little agitation of the glass as possible.

Vacuum Bubbles.—Another phenomenon peculiar to this stage is the formation of vacuum bubbles. These are caused by the premature "setting" of the top surface of the melt, thus enclosing the inner portion in a rigid envelope. On further cooling, the contraction of the interior greatly enlarges any small bubbles that may be present and may even draw in considerable quantities of air through cracks in the pot. This effect can be entirely prevented by insulating the top surface of the glass sufficiently to prevent the temperature of that portion from falling much below the temperature of the center of the melt.

Crystallization.—While the glass is yet soft, conditions are particularly favorable for the formation of crystalline phases in the melt, giving rise to the phenomenon of "crystallization" or "devitrification."[1] In addition, undissolved particles of sand or of the pot may form nuclei for the deposition of crystalline material from the glass. Given time enough at a suitable temperature, one or more crystalline phases may separate from any glass, but this effect begins reluctantly and proceeds slowly, so that most glasses can be cooled rapidly enough to prevent it.

Conditions to be Secured during Stage of Fluidity.—To inhibit convection and the formation of bubbles, the surface of the glass should be kept warm and heat removed chiefly from the bottom. The cooling should be made rapid down to a temperature below that at which there is no further danger of crystallization or devitrification. Convection can be further reduced by intensive chilling of the bottom of the pot by means of a current of compressed air or a fine stream of water, during the last hour before it is removed from the melting furnace.

Annealing Range.

The rate of cooling through the second stage determines the size and shape of the pieces into which the melt will break. The whole of the cooled melt, except the portion close to the pot, should be found broken into large, reasonably rectangular blocks, having smooth, flat surfaces through which their interior may be examined. Under these conditions the blocks have been

[1] N. L. Bowen, *J. Am. Ceram. Soc.*, 1, 599 (1918).

found to break clearly, but where the blocks have curved surfaces, they tend to form wedge-shaped fragments having very rough surfaces when broken. In extreme cases the interior portion of the melt may be found in the form of a large, flattened sphere of glass called a "marble" or "onion," which can seldom be broken satisfactorily.

When cold, the melt is usually found to be cut by cracks of two types: curved cracks, roughly spherical and concentric with the center of the melt, which form shells an inch or two thick in the outer portion of the melt; and plane cracks which divide the melt into large blocks.

Spherical Cracks.—If, as must always be the case, different parts of the melt cool at different rates, the resulting differences in rate of contraction cause stresses in the melt, which in turn tend to make the glass flow in such a way as to relieve the strains set up by them. The velocity of this release of strain decreases as the temperature falls, because of the increase of viscosity on cooling, and is, at any instant, nearly proportional to the total amount of strain causing it.[1] Consequently the amount of strain present will tend to approach such a value that it will be relieved, by internal flow, at the same rate as that at which fresh strain is introduced. As this flow is not very rapid, even at high temperatures, and is imperceptible at low temperatures, strain persists long after fresh strain ceases to be introduced.

Rather than consider the differences between cooling rates, it is somewhat simpler to speak of the time rate of change of the temperature drop (since the difference between the rates of change of two variables is equal to the rate of change of their difference, this amounts to the same thing). Then, if we assume that the coefficient of linear expansion of the glass is constant over the maximum temperature range throughout the melt at any instant, the differential rate of contraction, that is the time rate at which strain is introduced, is everywhere proportional to the time rate of change of the temperature gradient.

When the melt is set out of the melting furnace, there is little temperature difference through it. On account of convection

[1] This question is fully discussed in a paper in preparation by L. H. Adams and E. D. Williamson.

and, perhaps, the transfer of considerable quantities of heat through the glass by radiation as well as by conduction, the temperature drop between the center and the outside of the glass does not reach a maximum until the melt has cooled to a low red heat. An idea of this can be secured from Fig. 7, following.

After passing the maximum this temperature drop decreases, and disappears when the melt has cooled to constant temperature.

While the temperature drop is increasing the outer portions of the melt are contracting more rapidly than the inner and the strain consists of radial compression throughout, accompanied by tangential tension in the outer portions and tangential compression in the inner.[1]

After the maximum drop is passed, the inner portions are contracting more rapidly than the outer, and the stresses being introduced consist of radial tension throughout, accompanied by tangential compression in the outer portion and tangential tension in the inner. As the strains now being introduced are the reverse of those introduced before the temperature drop reached its maximum, their first effect is to neutralize the residuum of the strain introduced at that earlier time. On further cooling and reduction of the temperature drop strain builds up again and tends to fracture the glass by curved cracks more or less normal to the direction of the forces of radial tension.[2] During the earlier part of the annealing range, as has been said, strain is relieved by flow of the glass so much more rapidly than at lower temperatures that it does not accumulate sufficiently to cause cracks. Thus the spherical cracks are entirely due to the later decrease of temperature gradient.

This tendency to fracture increases as we recede from the center,[2] therefore fractures tend to follow one another, as strain increases, from the outside toward the center of the melt. If no other factor appears, the final form of the melt is a roughly

[1] We here assume that the isothermal surfaces in the melt are spheres. Actually they appear to be roughly spheroidal, approaching the spherical form at the center.

[2] The question of the elastic stresses produced by temperature differences in a solid sphere has been discussed by E. D. Williamson, *J. Wash. Acad. Sci.*, **9**, 209–217 (1919).

spherical core, or "marble," of glass surrounded by a series of shells very like those of an onion. The size of this core depends on the course of the temperature gradient.

This effect is shown in Fig. 1, which is a photograph of a melt

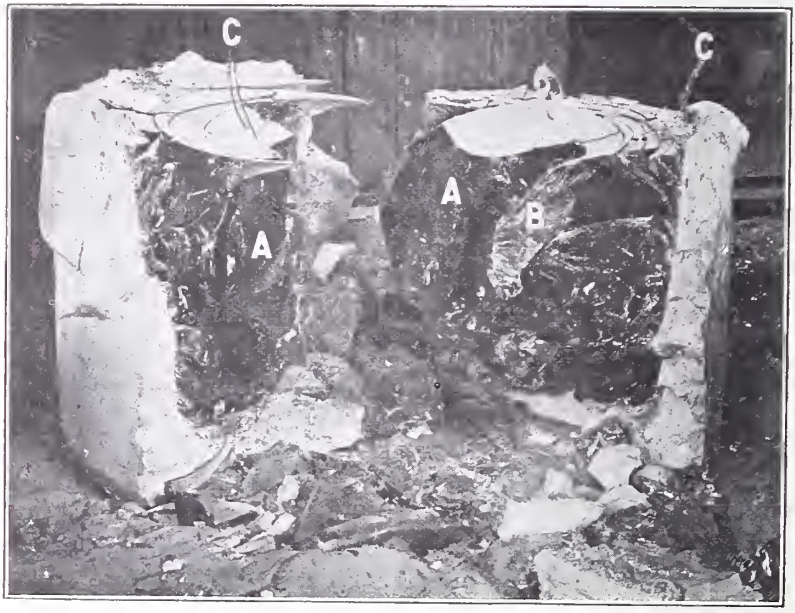


FIG. 1.—Spherical cracks in a poorly annealed melt.

that was cooled too rapidly. The cracks here are chiefly of the spherical type, appearing on the top surface as concentric circles. The inner core A (broken in the picture), was quite large, and as is usually the case, includes considerable of the top surface. The shape and irregular surface of the block B are characteristic of poorly annealed melts where the central core is not found unbroken. The wires C are thermocouples placed in the melt while it was still molten. This is an excellent example of a poorly cooled melt.

Marbles.—Where the central core remains unbroken the resulting marble is often so large as to include not only the top surface of the glass but some of the side walls of the pot as well.

When it is broken it yields blocks like B in Fig. 1; or, in extreme cases, the whole marble may be shattered by a very large number of radial cracks, as in Fig. 2. In this figure the center of the melt was at C, and S was the outside surface of the marble.

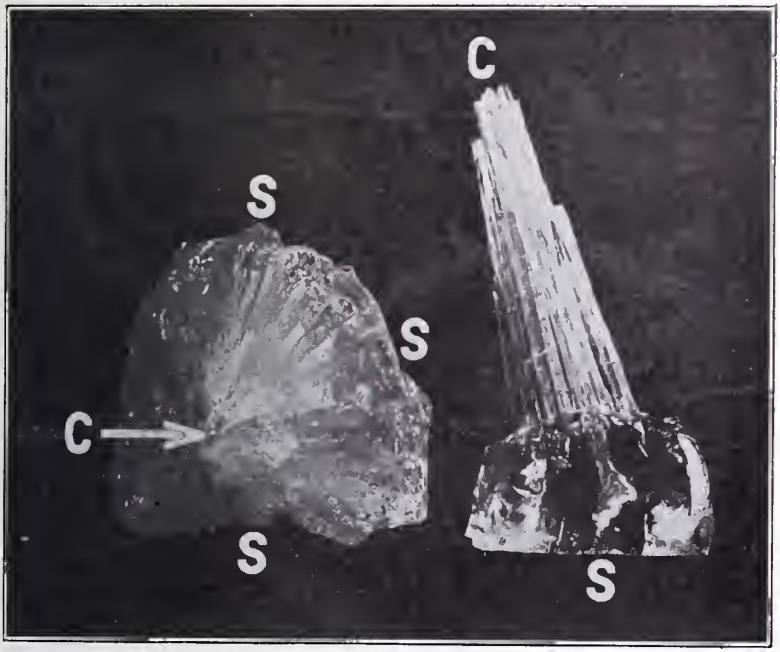


FIG. 2.—Block of glass obtained on breaking a heavily strained marble.

Plane Cracks.—When the melt has cooled to a temperature usually between 200° and 400° C, cracks start in the glass at the pot wall. These are probably due to the difference in contraction between the glass and the pot. These cracks extend into the melt until they reach another crack, or the opposite side of the pot. If the interior of the melt has not been separated from the pot by spherical cracks, the first plane crack to appear usually divides the melt in two along a vertical diametral plane. Subsequent cracks are usually not radial, but perpendicular to the first one. After a comparatively few cracks have formed,

the tendency for the cracks to follow the shortest path confines their activity to the outer portion of the melt. Here they cut the glass into pieces the size of one's fist or smaller. Horizontal cracks form as well as vertical, but are usually subordinate to the vertical cracks.

The presence of these cracks in the interior not only prevents the formation of a marble by cutting it up before it is formed but also greatly reduces the amount of strain present in the interior.

Fig. 3 is a photograph of a well annealed melt, broken in two

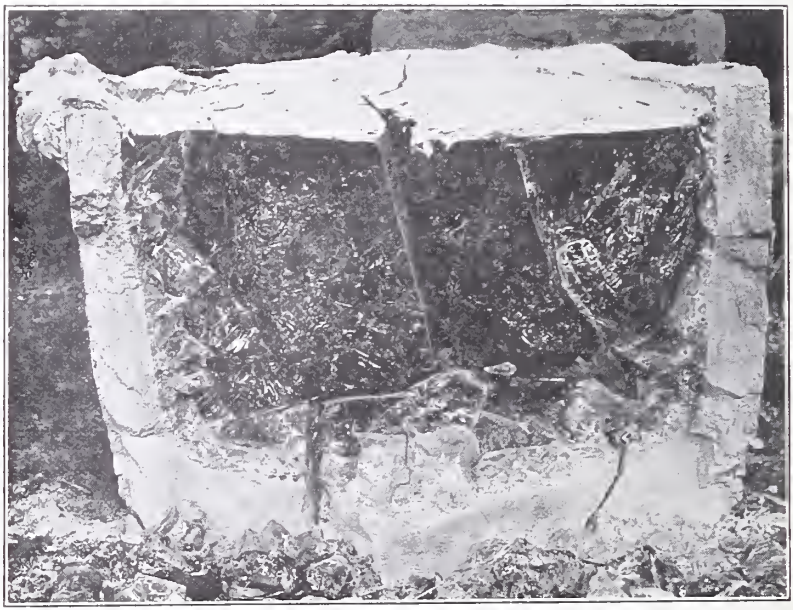


Fig. 3.—Plane cracks in a well-annealed melt.

along the diametral plane crack. The cracks here are entirely of the plane type, the surfaces are smooth and the blocks of glass are large. The smaller fragments are confined to the bottom and sides of the melt where the glass is certain to be full of striae dissolved from the pot.

An experiment was carried out to show the effect of the pres-

ence of plane cracks on the strain in the melt, and to prove that they were caused by the pot. A "marble" about 20 inches (50 cm.) in diameter was insulated with a thick layer of sand and heated very slowly to its annealing temperature. It was then cooled to room temperature at a rate somewhat slower than the rate at which 36-inch (90 cm.) melts were cooled. Neither spherical nor plane cracks appeared on cooling. On being cut up with a cold chisel, the marble broke into large pieces, but of irregular shape with rather rough surfaces. 36-inch (90 cm.) pots of glass, although cooled more rapidly than the above, showed plane cracks with smooth surfaces.

The temperature at which the formation of plane cracks begins seems to be nearly independent of the temperature gradient and cooling rate. Therefore it is possible, by controlling the temperature gradient, to hold back the formation of spherical cracks until the plane fracturing is well under way.

Temperature Limits of Annealing Range.—We are now in a position to define the temperature limits of the annealing range, and, from them, those of the other two stages.

If a small block of glass, placed in a polariscope, is heated rapidly, it will exhibit the characteristic colored figure of glass under strain. When the block is passing through a certain temperature range the color changes rather rapidly. We shall call the average temperature of the block at this period the "annealing temperature."[1] It depends on the size of the block and on the rate of heating, but does not vary more than 20°, provided the smallest dimension of the block is less than 1 cm. and that it is heated at a rate nearly rapid enough to crack it. The greater part of the strain present in a block of glass held at this temperature will vanish in a few seconds. Thus the extent to which strain can accumulate at this temperature is small compared with the rate at which it is introduced.

This temperature can be more definitely fixed as that at which a given proportion of the strain in a block of a certain size will disappear in a given time. For our purpose a rougher, more easily determined point is sufficient, and preferable.

[1] The "annealing temperature" determined by this method is considerably higher than the temperature usually employed in the annealing kilns.

The "annealing temperature" of the glass is here taken as the upper limit of the "annealing range" of the melt (see page 544). The lower limit is taken as the temperature at which the formation of plane cracks begins.

The lower limiting temperature depends on the relation of the coefficient of expansion of the glass to that of the pot. As a first approximation it may be assumed to be 250° to 300° below the annealing temperature.

Conditions to be Secured through Annealing Range.—As the spherical cracks are entirely due to the reduction of the temperature gradient, it is best to keep this low in the first place, and then to prevent its decreasing greatly until the formation of plane cracks is well under way. These conditions can be attained by decreasing the cooling rate at a time shortly before the annealing temperature is reached, by adding insulation or otherwise; and by removing the insulation a little while before the end of the annealing range. Removing the insulation will usually increase the temperature gradient temporarily, thereby neutralizing some of the strain present and gaining time for the plane cracks to appear.

Methods of Retarding the Cooling.

The rate of cooling through the annealing range may be retarded by placing the pot in an inclosed space, such as a kiln or pot-arch, and heating the air around it; or by surrounding the pot with a layer of insulating material. Either method gives a satisfactory result, and there seems to be no great advantage in combining them, except that in any case the surface of the glass should not be exposed, but covered by a layer of insulating material having at least the insulating properties of the pot wall itself.

Either of these methods depends on the fact that the melt tends to cool to the temperature of its surroundings at a rate that is more or less proportional to the difference between its temperature and that of its surroundings. Whether the melt is insulated by the addition of some covering or by the presence of the pot alone, the ratio of cooling rate to temperature difference decreases with increase of insulation. Thus a given rate

of cooling at any temperature may be attained by adding sufficient insulation at room temperature, or by placing the melt in a sufficiently heated enclosure, with no insulation beside that afforded by the pot and the layer of insulation previously applied to the surface of the glass.

Use of a Heated Kiln or Pot-Arch.—In this method the rate of cooling can be controlled by varying the temperature or the rate at which air is supplied to the pot. Two general methods have been developed: (1) where the temperature and rate are kept nearly constant; and (2), where the temperature and rate are decreased as the melt cools.

In Fig. 4, A, B, and C are time-temperature curves for a 36-inch

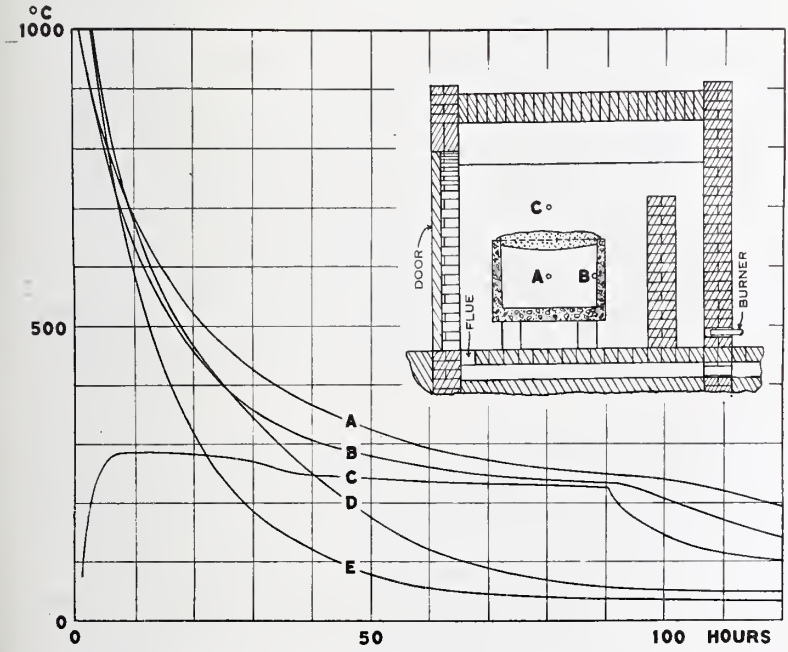


FIG. 4.—Time-temperature curves for melt cooling without added insulation. Curves A, B, and C apply to a melt cooled in a heated pot-arch; Curves D and E to a melt cooled in the open.

(90 cm.) pot of light barium crown glass, (n_D = 1.57) annealed in a heated pot-arch. Curve A represents the temperature at

the center of the melt; Curve B that at a point on the inside wall of the pot, midway between top and bottom; Curve C that of the air 8 inches (20 cm.) above the pot. The top surface of the glass was covered with a 4-inch (10 cm.) layer of kieselguhr immediately after setting the melt out of the melting furnace. About an hour later the gas was lighted in the arch. Zero time in Fig. 4 and subsequent figures is the time at which the pot was set out of the melting furnace.

Curves D and E in the same figure are for a 36-inch pot of medium flint glass ($n_D = 1.61$), insulated on the surface as before, but allowed to cool in the open at a temperature of about 30°. Curve D represents the temperature at the center of the melt and Curve E that at a point in contact with the inside of the pot wall midway between top and bottom. In both cases the temperatures were measured by bare-wire thermoelements permanently placed with their junctions at the points indicated.

As the two glasses used in these experiments have not been found to differ widely in their thermal properties, these two sets of curves are comparable and show what effect is to be expected when the temperature to which the melt can cool is raised.

Curves A and B are not only less steep than D and E, but closer together, indicating a smaller temperature difference. At the time the gas is shut off in the arch, they begin to diverge again, thus preventing the temperature difference from decreasing below its former minimum until the melt reaches a low temperature.

In the case of Curves A, B, and C, no attempt was made to cool the melt rapidly through the stage of fluidity. In practice it would be better to let it cool 3 or 4 hours in the open before it is placed in the arch; and to have the arch heated while the pot is waiting rather than after it goes in.

This method was developed with the expectation of building a long, heated tunnel through which the pots should be moved on cars; the length of the tunnel to be such that it should contain three days' run of pots under conditions of maximum production. It was found that if a slow stream of air were supplied to the tunnel at a constant temperature of 300° C, the annealing would be entirely satisfactory.

Dr. C. N. Fenner, of the Geophysical Laboratory, developed the following method at the plant of the Spencer Lens Co.: When a melt was removed from the melting furnace, it was allowed to cool freely until a new pot had been taken from an individual pot arch and set into the melting furnace. The melt, having cooled about 15 minutes in the open and until convection currents had subsided, was then placed in the hot pot-arch from which the new pot had been taken. The burners of the arch were then adjusted so that its temperature would fall to a low red heat (500 to 600°) over night (about 16 hours). He then found that the melt would anneal well: (1) if the firing were adjusted so that the temperature of the air would reach about 400° at the end of another 24 hours, or (2) if the flame were turned completely out. In the latter case, since both the firing ports and flues of the arch were close to the bottom, there was probably no tendency for air to circulate through it, and it cooled solely by the conduction of heat through the walls of the pot-arch. If, however, the flame was turned very low, but not out, a circulation of relatively cool air was induced which caused the melt to cool so rapidly that it was poorly annealed.

No data are available for the temperatures within the melt, but it seems likely that this procedure causes the temperature gradient to remain small, and to reach its maximum at a temperature well within the annealing range. Thus the strains that fracture the melt did not begin to appear until a low temperature was reached.

At the Bausch & Lomb plant, C. N. Fenner and R. E. Hall developed a method similar to that which was later used at the Spencer Lens plant, except that fire was maintained in the pot arch until it had reached a temperature of 200° to 300°. This method also probably keeps the temperature gradient low, and increases it again just after the melt has passed out of the annealing range.

These three methods are summarized in the curves of Fig. 5, which represent the temperatures of the air over or close to the pot, plotted against the elapsed time after setting the melt out of the melting furnace. The curves are in three sets. Each set consists of a solid, a dashed, and a dotted curve, showing the

temperature regulation which resulted in good, fair, and poor annealing, respectively, for each of the three methods described below (Pittsburgh, Spencer, and Bausch & Lomb). These curves were made on various types of glass and sizes of pots, and the character of the annealing was judged by three different men at three different plants. Thus the three sets cannot be directly compared.

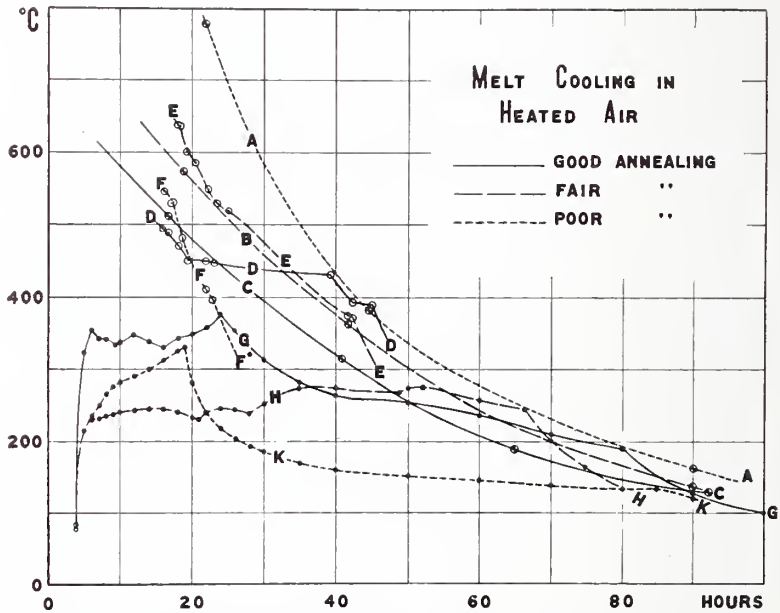


FIG. 5.—Time-temperature curves for melt cooling in heated air. The cooling was retarded by placing the pot in a heated pot-arch. The temperatures are those of the air over, and close to the pot.

Curves A, B, and C are plotted from data on the temperature of the air above the pot, taken at the Bausch & Lomb plant, where the fire was kept burning in the arch until it had cooled to a low temperature. Each of these three curves was plotted from 3 (in one case 4) measurements, which accounts for their marked regularity. In spite of the fact that the lowest average cooling rate during the first three days was in the case of A, nevertheless A gave the poorest annealing. Here the arch was

kept for a long time at a high temperature, which greatly reduced the temperature gradient at this temperature, but did not prevent its reaching a high value as the melt passed rapidly through the annealing range. In the case of B and C, the melt cooled rapidly at high temperatures, but the cooling was greatly retarded through the annealing range.

Curves D, E, and F represent data taken at the Spencer Lens plant where the gas was left burning until the arch had cooled to about 400°. This set also illustrates the fact that early retardation is not important in securing satisfactory annealing.

The data for Curves G, H, and K were taken at the Charleroi plant of the Pittsburgh Plate Glass Co.; they are part of the same series of experiments as Curves A, B, and C of Fig. 4. In the case of G, while the annealing was entirely satisfactory, it was found later that the rate of cooling was slower than necessary. In the case of H, a somewhat slower rate seemed desirable at the start. Where the curve was more nearly the mean of G and H, it was found that a 36-inch melt could be removed from the arch at the end of $2^1/_2$ days, and broken at the end of 4 days. Curve K resulted, as was expected, in a marble. This schedule was designed to apply alike to all of the several types of glass made at that plant, and could therefore be somewhat shortened for individual cases.

Use of Insulation around the Melt.—Insulation may be applied to the pot either in the form of an insulating cover, or the pot may be surrounded by a layer of some loose insulating material such as sand or kieselguhr. Where sand is used, a thickness of 8 inches (20 cm.) is sufficient for pots 30 to 50 inches (75 to 120 cm.) in diameter. This may be reduced, in the case of kieselguhr, to 3 inches ($7^1/_2$ cm.).

The form of cover used at the Pittsburgh Plate Glass Company's plant consisted of a hollow double-walled sheet iron cylinder, closed at one end by a double wall. The 3-inch ($7^1/_2$ cm.) space between the walls was filled with kieselguhr, and two handles were riveted to the outside to facilitate handling. The height inside the cover was such that, when it was inverted over a pot, the bottom edge of the cover rested on the floor, and could be

sealed with a little sand. The weight of such a cover for a 36-inch pot (90 cm.) was perhaps 150 lb. (68 kg.).

At the same plant, previous to the use of these covers, the melts were insulated with sand, held in place by a retaining case of sheet iron. These cases were made by riveting together two sheets of flat galvanized iron to form a continuous band. Their average diameter was greater than that of the pot, by 16 inches (40 cm.) or more, and their height was 30 inches (75 cm.). This height brought the top of the case to about the same level as the surface of the glass in the pot.

These cases were filled by shoveling the sand into them, an operation that consumed considerable time, as it involved moving about a ton of sand for each 36-inch pot. When the sand was to be removed from the pot, the case was first lifted off by the crane, allowing the sand to form a cone around the bottom of the pot. This cone was then hoed back to clear the pot.

Of the two, the portable covers were preferable, entailing less time and labor and being more tidy. They were objected to at first on the ground that circulation in the air space between the pot and the cover might induce too rapid cooling. This was found not to be the case.

In Fig. 6 time-temperature curves are plotted for the cooling of two melts of medium flint glass ($n_D = 1.61$) in 36-inch (90 cm.) pots. In the case of Curves A, B, and C, the melt cooled freely for 5 hours after it was set out of the furnace; it was then insulated with a 10-inch (25 cm.) layer of sand retained by a sheet-iron case; this insulation was removed 62 hours after setting out. Curve A represents the temperature at the center of the melt; Curve B, that at a point in contact with the inside of the pot midway between top and bottom; and Curve C, that on the outside of the pot opposite point B. The melt, sand and retaining case are drawn to scale in the upper part of the figure, and the location of the thermocouples shown by small circles. In the case of Curves D and E, the melt was cooled in the open with no insulation other than a 4-inch (10 cm.) layer of kieselguhr on top. Curve D is for a point in the melt corresponding to that of Curve A; Curve E, for a point corresponding to that of Curve E. These two curves are the Curves D and E of Fig. 4.

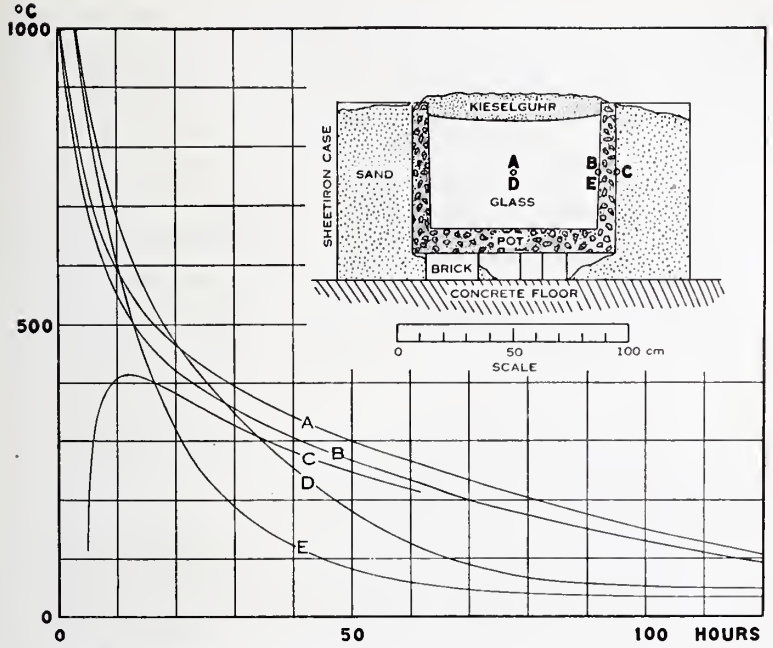


FIG. 6.—Time-temperature curves for a melt cooled in sand. Curves D and E of Fig. 4 are reproduced for comparison.

Course of the Temperature Drop.—In Fig. 7 the difference in temperature between the center and side of the melt, for the three

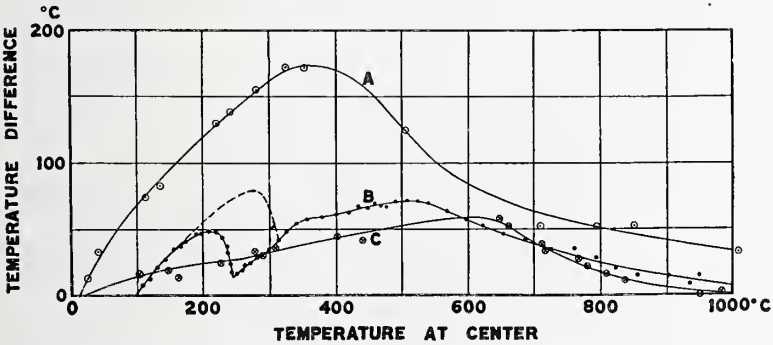


FIG. 7.—Temperature drop through the melt plotted against the corresponding temperature at the center, for the three melts of Fig. 4 and Fig. 6.

melts of Fig. 4 and Fig. 6, are plotted against the corresponding temperatures at the center. Curve A is for the melt cooled in the open (D and E of both Fig. 4 and Fig. 6); Curve B, for that cooled in a pot arch (A, B, and C of Fig. 4); Curve C, for that cooled in sand (A, B, and C of Fig. 6).

Most of the cracks in melt A were of the plane type, and there were many fragments weighing 10 lbs. ($4^1/_2$ kg.) or over. This may be explained by the fact that the temperature difference did not begin to increase until the outside of the melt had cooled to about 200°, a temperature well below that at which plane fracturing usually begins in this type of melt. However, the break could not be considered as satisfactory because of the large proportion of glass found broken into small fragments.

Curve B shows two maxima. The first, at about 510°, was only attained after the center had cooled through more than 500° at high temperatures; the second, at about 210°, was reached on shutting off the gas in the arch after the center had cooled through only 40°. This shows the very great role played by convection and radiation in reducing the temperature drop. The portion of the curve to the left of 250° gives an idea of the extent to which the fall of the temperature drop can be postponed. In later experiments the gas was turned out in the arch much sooner, which would have caused the curve in this case to take a form something like that indicated by the dotted line in the figure. This practice resulted in as good a break and a very much shortened time of cooling.

The lower portion, below 260°, of Curve C shows a much smaller break than that of Curve B, probably because the cone of sand was not completely removed from that side of the pot—an experimental detail that was unfortunately not recorded.

Cooling Schedule, where Insulation is Applied to the Pot.—The following schedule was developed by the author at the Charleroi plant of the Pittsburgh Plate Glass Company for cooling their 36-inch (90 cm.) pots and 49-inch (125 cm.) pots. As each type of glass was set out of the melting furnace at its definite temperature, the operations of cooling were performed according to the time that had elapsed after the pot was set out. The

schedule was so arranged that all pots of the same size were treated alike, regardless of the type of glass they contained.

Immediately on setting the melt out of the furnace, the surface of the glass was covered with a 4-inch (10 cm.) layer of kieselguhr and the pot placed on three bricks to cool.

Four hours later in the case of 36-inch pots, and eight hours in the case of 49-inch pots, insulation was applied to the pot in the form of an 8-inch (20 cm.) layer of loose sand or in the form of one of the covers described above.

The insulation was removed from the 36-inch pots 3 days after setting out; and from the 49-inch pots 5 days after setting out. Two or three days later the melts were cool enough to be broken down.

Where 36-inch melts are cooled in sand according to this schedule the very approximate temperatures given in Table 1 may be expected at the center of the melt. The third and fifth columns of numbers refer to the times insulation is applied and removed, respectively. Some of the annealing temperatures given in the fourth column were determined by G. W. Morey, of the Geophysical Laboratory, using the rough method given above; the others were extrapolated from data obtained for the release of strain at lower temperatures by L. H. Adams and E. D. Williamson, of this laboratory.

TABLE 1.

Approximate Temperatures at Center of 36-inch Melts Cooled in Sand.

Type of glass.	n_D.	Temp. set out.	Temp. after 4 hrs.	Annealing temp.	Temp. after 72 hrs.
Flint.....	1.57	1038°	800°	465°	320°
Flint.....	1.61	996	780	455	300
Flint.....	1.66	968	760	445	290
Crown, Ordinary.....	1.52	1093	850	570	350
Crown, Borosilicate....	1.52	1116	860	590	360
Crown, Barium.....	1.57	1016	790	580	310
Crown, Barium.....	1.61	1016	790	600	310

Breaking Down.—When the melt is ready to be broken down, the diametral crack in the glass is first located, and the pot broken

at the ends of it with a sledge. The two halves of the melt are now separated and the blocks of glass loosened by beating on the outside of the pot, or shaking it with a crow bar. A small hammer is used where necessary to free the blocks of adhering pot shell.

Summary.

The conditions to be attained when a melt of optical glass is cooled in the pot are: (1) that neither ream nor bubbles shall be introduced during the cooling, nor carried into the middle of the melt; (2) that the glass shall not become inhomogeneous through the precipitation of a crystalline phase; (3) that the bulk of the cooled melt shall be found cracked into large, reasonably rectangular blocks, having smooth, flat surfaces; and (4) that these blocks shall be sufficiently free from strain to cleave readily with a smooth fracture.

The appearance of ream in the middle of the melt, vacuum bubbles and a crystalline phase, can be discouraged by rapid cooling, preferably from the bottom of the pot, while the glass is still soft; and by insulating its top surface as soon as the melt is set out of the melting furnace.

The cold melt shows cracks of two types: "spherical cracks," and "plane cracks." The spherical cracks are due to decrease of temperature difference, and first appear below the temperature at which the drop is a maximum; these cracks form one after another until the temperature gradient has disappeared. The plane cracks seem to be the result of a difference in rate of contraction between the glass and the pot; the temperature at which they first appear in a given melt seems to be nearly independent of the cooling rate. The presence of either type of crack in the melt reduces the tendency of the other type of crack to form. As the fracture due to spherical cracks is rough and the pieces formed are irregular, while that due to plane cracks is entirely satisfactory, it is desirable (1) to maintain the temperature gradient at a low value by slow cooling; and (2) to prevent its decreasing much below its maximum until after the formation of plane cracks has begun, by increasing the cooling rate at the proper time.

The cooling rate can be reduced (1) by heating the surroundings of the melt, *i. e.*, placing it in a heated kiln; and (2) by surrounding it with an envelop of some such insulating material as sand, kieselguhr, etc. Variations of these two methods are described and time-temperature data given.

GEOPHYSICAL LABORATORY,
CARNEGIE INSTITUTION OF WASHINGTON,
April, 1919.

SPECIAL SPARK-PLUG PORCELAINS.[1]

By A. V. Bleininger and F. H. Riddle.

Requisite Properties of Porcelains for Spark Plugs.—The average commercial porcelain does not fulfill the conditions required for spark-plug service, nor for any other conditions where high-tension currents are employed and the temperature is considerably above atmospheric conditions. From the standpoint of the electrical resistance at the temperatures reached in airplane engines, the feldspathic porcelains begin to break down at a rate rapidly increasing with temperature, due to electrolytic effects. This is indicated also by the polarization which is observed in using direct current. The leakage thus taking place may become a serious factor.

The average porcelain is likewise not constant in volume and the volumetric changes are greater than can be explained solely by thermal expansion. Aside from the thermal expansion, the quartz content of porcelain, when heated or cooled, is subject to certain modifications in crystalline structure, accompanied by definite volume changes. Thus, the form of quartz permanent at atmospheric temperature, is alpha quartz which inverts to beta quartz at 570° C, a transformation which is reversed by cooling. On the other hand, if, in the firing of porcelain, the quartz is inverted to its high-temperature form, cristobalite, there is again the transformation of the alpha to the beta modification to be considered, which takes place at 230° C. In either case, volume changes are unavoidable in all porcelains containing free quartz in large amounts, due to the inversion noted. In a good spark-plug porcelain, the quartz should be eliminated from the composition and replaced by a substance not subject to these inversions.

Another defect of the ordinary porcelain is that the coefficient of thermal expansion is by no means constant at different tem

[1] By permission of the Director, Bureau of Standards.

peratures. It may be 19 × 10^{-6} between 30–200° C and 5 × 10^{-6} between 400–500° C.

With respect to mechanical strength, also, great variations are possible. A series of tests of commercial electrical porcelains, conducted at the Pittsburgh laboratory of the Bureau of Standards, showed variations in the modulus of elasticity of from 1,600,000 to 6,000,000.

Methods of Testing Spark-Plug Bodies.—For the purpose of studying porcelains from the standpoint of their electrical conductivity at the high temperatures obtained in airplane ignition systems, a large number of typical compositions were made up in the form of test cups of 60 mm. outside diameter and 65 mm. high, the thickness being 2.5 mm. These mixtures were usually prepared in 10 kg. batches, carefully weighed out and ground wet in porcelain-lined ball mills for three hours. The suspension of clay was pumped into a filter press to remove the water and then kneaded in a mixing machine to the consistency required for shaping the mass on the potters wheel. This was done by the use of plaster molds in which the cups were "jiggered" in the usual manner. The specimens were then dried, placed in saggers and burned in a down-draft kiln fired with natural gas to the finishing temperature. The latter was controlled both by means of thermocouples with the necessary galvanometers, and pyrometric cones. Each cup was examined for non-absorption by the application of ink. The testing of the specimens was done at the Washington laboratory. The characteristic expression for the resistivity of the porcelains is the "Te" value, which represents the temperature in degrees C at which a cubic centimeter of the material still shows a resistance of one megohm.[1]

Replacement of Feldspar.—Upon comparing the "Te" value of the different porcelains it was noted that there exists no definite relation between the composition and the electrical conductivity. On the other hand, the higher the maturing temperatures of the porcelains, the higher was the "Te" value. This is practically equivalent to saying that, roughly speaking, the electrical resistance at higher temperatures is the greater, the lower the feldspar content, since small amounts of this flux make it neces-

[1] *J. Am. Ceram. Soc.*, 1, 700 (1918).

sary to carry the porcelain to a higher burning temperature. The relation, however, is not sharply defined, as may be observed from the results compiled in the following table:

TABLE I.

No. of body.	Per cent feldspar.	Maturing temperature in cones.	"Te" value.
16	16	16	560
17	18	15	390
18	20	14	440
22	28	14 (1/2 over)	370
23	30	13	450

It is quite evident that the micro-structure of the porcelains is an important factor in this connection, since it cannot be immaterial how much kaolin or quartz has been dissolved by the fused feldspar and how much sillimanite has been formed. The evidence, however, was considered sufficient to warrant the replacement of the feldspars by other fluxes, the oxides of magnesium and beryllium being used for this purpose.

Owing to the evolution of carbon dioxide from magnesite and the artificially prepared basic magnesium carbonate during the firing process, and the very large shrinkage accompanying their use as a flux, it was decided to introduce the magnesia in the form of a calcine; that is, a silicate mixture previously fired tovitrification. The mixtures employed for this purpose corresponded to the formulas: $MgO.Al_2O_3.2SiO_2$; $MgO.Al_2O_3.4SiO_2$ and $MgO.Al_2O_3.6SiO_2$. Of these, the first and the second were most used in this work. The preparation of the calcines consisted in dry ball-mill-grinding and firing the mixtures made up into balls with just sufficient water. After calcination, the material was crushed and ground and introduced into the bodies in this form. The beryllium oxide was brought in through the use of the mineral beryl (which has the general composition $3BeO.Al_2O_3.6SiO_2$) without any previous heat treatment.

With the use of magnesia as the principal flux the electrical resistance, and hence the value "Te," was found to increase quite decidedly, though with no well defined regularity as referred to

percentage content of magnesia. It was seen again, in this connection, that the structure of the porcelain plays an important part especially as it is known that MgO accelerates the crystallization of sillimanite most vigorously. Not only the number but also the size of the sillimanite crystals is of significance in determining the texture of the body, whether it is to be fine-grained, glassy, or coarsely crystalline. The rate of cooling the porcelain is likewise of importance since a rapid drop in temperature invariably causes the structure to be closer and of more vitreous character than when a longer time is taken in cooling down the kiln. For this reason smaller kilns are to be preferred to larger ones since they permit of more rapid cooling. The effect of magnesia, added in the form of synthetic silicate, is strikingly shown by the high "Te" values of bodies Nos. 77 and 78 (see Table 4).

It is a fact that the magnesian silicates show electrolytic effects which are much less prominent than when feldspar is used as a flux. It likewise appears that the higher the firing temperature of the porcelain the greater its electrical resistance, at temperatures up to 700° C, or somewhat above this point.

With reference to the use of beryllium oxide, it was found that this flux behaves similarly to the magnesia in showing high electrical resistance and "Te" values. This is indicated by the following table:

TABLE 2.

No.	Per cent beryl.	Per cent clay.	Per cent flint.	Maturing temp. of porcelain in cones.	"Te" value.
193.....	25	50	25	12	624
194.....	35	50	15	11	784
195.....	45	50	5	11	798

It is evident from these results that beryllium oxide, used in the form of the mineral beryl, is a valuable flux from the standpoint here under consideration. It requires, however, careful temperature control in firing, since the beryllium porcelain is subject to sudden deformation as the vitrification temperature is exceeded. The experiments have proven that beryllium oxide

is worthy of consideration for the production of such porcelain, and the high "Te" value obtained, 798, is exceedingly promising. The firing temperature of these bodies is quite low, from cones 11 to 12.

Another interesting fact was that the thermal expansion of the beryllium porcelain was found to be lower than that of the feldspathic bodies commonly employed. The average coefficient was found to be 1.63×10^{-6} for the temperature interval 26° to 200° C; 2.95×10^{-6} for 200° to 400°; 3.60×10^{-6} for 400° to 570°; and 2.33×10^{-6} for 26° to 400°.

Replacement of Quartz.—From a general study of porcelains, it appears desirable to eliminate the quartz, as has been pointed out in a previous paragraph. Some of the materials available for this purpose are calcined kaolin, synthetically prepared sillimanite ($Al_2O_3.SiO_2$), sintered or fused alumina, and zirconium oxide. These substances have been introduced in a number of compositions. The effect of adding calcined kaolin in general was beneficial; even with a feldspar content of 13.5 per cent (body No. 63) a fair "Te" value, 540, was obtained. This particular composition contained 50 per cent raw kaolin, 13.5 per cent feldspar, 1.5 per cent calcium carbonate, and 35 per cent calcined kaolin. The maturing temperature of this porcelain was that corresponding to cone 16. It is evident that by raising the content of calcined kaolin still more at the expense of the feldspar a higher "Te" value would be obtained. The introduction of plastic ball clay to replace kaolin, invariably and in all types of bodies, reduces the electrical resistance within the temperature range here under consideration.

The use of fused alumina in any extensive work was prohibited by the lack of material low in iron content. The commercial substance is unsuited for this purpose. The total amount of white, fused alumina available was not more than two pounds. To bring about the necessary impervious and dense structure the use of 17.6 per cent of feldspar was required. A mixture consisting of 45 per cent kaolin, 17.6 per cent feldspar, 4.4 per cent calcine No. 13, and 33 per cent fused alumina, resulted in a very tough porcelain which showed a "Te" value of 620. Cal-

cine No. 13 was compounded, according to the formula $MgO.Al_2O_3.2SiO_2$, from 84 parts, by weight, of magnesium carbonate, and 258 parts of plastic kaolin from Florida and Georgia. Here again the reduction of the feldspar content and its replacement by calcine No. 13, or beryl, or a combination of these two, would be certain to raise the "Te" value considerably and at the same time would result in a porcelain of excellent mechanical properties. The cost of the fused, white alumina would be quite high, but would be justified under the circumstances. Furthermore, a single calcine could be readily produced by combining the raw materials of calcine No. 13, the kaolin and magnesite, with uncalcined alumina from any convenient source, and firing the mixture to a point of constant volume, which could be accomplished at temperatures not exceeding cone 18. At the same time this procedure would simplify the process of preparation very considerably.

Sillimanite ($Al_2O_3.SiO_2$) is a normal component of all hard-fired porcelains, and this constituent, if not present in the form of large crystals, imparts to the material constancy in volume upon heating, lowers the thermal expansion, and increases the refractoriness and the resistance to sudden heating and cooling. For this reason it was thought desirable to produce the mineral synthetically and to introduce it in the porcelain composition in place of the quartz. This was done by combining 70.2 per cent of plastic kaolin, 27.8 per cent anhydrous alumina, and 2 per cent boric acid. After grinding this mixture in the dry state in a ball mill, it was made up with water to form a plastic mass which was molded into balls which were calcined to a temperature corresponding to that of cone 20, or approximately 1530° C. At this temperature the mass sintered to a dense structure, subject to but little additional shrinkage and containing only a small amount of uncombined alumina. The boric acid was added to assist in bringing about the necessary shrinkage and closing up of the pores, and it is quite probable that most of this constituent is volatilized at the final temperature of calcination. This calcine (No. 19) was then crushed separately, passed over a magnetic separator and then ground together with the other materials of the body with the addition of water, in the ball mill. In preparing

this type of body the feldspar was eliminated, on the basis of the results discussed above, and replaced by a calcine corresponding to the formula $MgO.Al_2O_3.4SiO_2$ (No. 14). This porcelain (No. 152) was composed of 30 per cent kaolin, 10 per cent ball clay, 40 per cent sillimanite calcine and 20 per cent fluxing calcine No. 14, and fired to a temperature of cone 16 or higher. The resultant material had excellent electrical qualities and an average "Te" value of 690. The structure of this body should be fine and dense, a condition which requires quite rapid cooling of the kiln. Slower cooling results in the formation of coarser sillimanite crystals which produce a structure much less resistant to sudden heating and cooling.

The preparation of this body may be simplified by the combination of the two calcines into one. Thus, by employing the composition of flux No. 13 ($MgO.Al_2O_3.2SiO_2$) the combined calcine would be composed of 75.10 per cent plastic kaolin, 8.77 magnesite, and 16.13 per cent anhydrous alumina. The boric acid content has been eliminated. Body No. 152 would then consist of 30 per cent kaolin, 10 per cent ball clay, and 60 per cent of the combined calcine. Wherever the working conditions permit, the ball clay should be eliminated, and an effort made to employ the body containing 40 per cent of plastic kaolin, which can usually be done by allowing the mixture to age before molding it.

The tendency of the body to crystallize may be diminished also, by replacing the fluxing calcine in part by beryl, so that these two components are present in the ratio of 1 to 1. If, then, the combined fluxing- and sillimanite-calcine is employed, the resultant composition would be: kaolin 60.94 per cent, anhydrous alumina 18.38 per cent, boric acid 1.32 per cent, magnesite 4.58 per cent, and beryl 14.78 per cent. The body, as before, would consist of 60 per cent of this calcine and 40 per cent of kaolin. The fusion point of this mixture is so low that it would be quite possible to eliminate the boric acid entirely. At the same time the vitrification point of the body is lower and closer to the normal kiln-temperatures.

Attention might be called to the fact that the use of a silicious porcelain is not objectionable from the electrical standpoint, but only from consideration of the mechanical strength, resistance

to sudden temperature changes, etc. This is shown by the results upon high silica porcelains, bodies Nos. 116 and 119, which show a very high "Te" value, namely 730. This type of porcelain contains 50 per cent clay, 30 per cent free quartz (flint) and 20 per cent magnesia calcine (Table 3). In No. 116, the calcine corresponds to the formula $MgO.Al_2O_3.2SiO_2$, and in No. 119 to $MgO.Al_2O_3.4SiO_2$. It is fair to state that these porcelains would give excellent results, considered from the dielectric standpoint, when used under conditions not so extreme with reference to temperature and shock—as is the case with spark plugs used in airplane engines.

Zirconium oxide was used only in two porcelains, due to the comparatively limited supply available. The material at hand was zircon which contained 52.74 per cent of ZrO_2 and 43.46 per cent of silica. As received, the mineral was high in iron, which, however, was eliminated by treatment with chlorine[1] at a temperature of 800° C. In this manner all but a trace of the iron was removed. Owing to the fact that these zircon porcelains were produced comparatively early in the work, feldspar was employed as the flux. The result was that the electrical resistance, and hence the "Te"

TABLE 3.

Body No.	Per cent clay.	Per cent quartz (flint.)	Per cent MgO-calcine.	Formula of MgO-calcine.	Sillimanite calcine, per cent.	Coefficient of thermal expansion x 10^{-6}. Degrees centigrade. 30–200°.	200–400°.	400–550°.	30–400°.
114	50	20	30	$MgO.Al_2O_3$-$6SiO_2$	..	19.45	9.35	5.52	13.99
116	50	30	20	$MgO.Al_2O_3$-$2SiO_2$	..	7.34	6.11	4.68	6.68
117	50	20	30	$MgO.Al_2O_3$-$2SiO_2$	..	8.93	4.43	4.05	6.51
119	50	30	20	$MgO.Al_2O_3$-$4SiO_2$	..	19.61	11.13	8.08	15.03
120	50	20	30	$MgO.Al_2O_3$-$4SiO_2$	..	10.45	5.43	4.45	7.74
152	40	..	20	$MgO.Al_2O_3$-$4SiO_2$	40	3.36	4.19	4.78	3.81

[1] A. J. Phillips, *J. Am. Ceram. Soc.*, 1, 791 (1918).

value, was low, being 500 and 450, respectively. With the use of the magnesia- or beryllium-fluxes much better products could undoubtedly be produced. The mechanical strength of these porcelains was excellent in every respect. The composition of these bodies, Nos. 72 and 73, is given in Table 4.

The effect of the quartz in porcelain upon the thermal expansion of the body is shown in the results compiled in Table 3.

TABLE 4.

Number of body.	16 %	17 %	18 %	22 %	23 %	24 %	28 %	32 %	35 %	36 %	39 %	40 %
Kaolin	45.00	45.00	45.00	45.00	45.00	50.00	50.00	55.00	55.00	55.00	55.00	60.00
Feldspar	16.00	18.00	20.00	28.00	30.00	16.00	24.00	14.50	20.50	22.50	28.50	16.00
Potters flint	39.00	37.00	35.00	27.00	25.00	34.00	26.00	29.00	23.00	21.00	15.00	24.00
Whiting-($CaCO_3$)	...	...	...	...	...	...	...	1.50	1.50	1.50	1.50	...
Ball clay	...	...	...	...	...	...	...	...	...	...	...	...
Calcined-kaolin	...	...	...	...	...	...	...	...	...	...	...	...
Calcine No. 3	...	...	...	...	...	...	...	...	...	...	...	...
Calcine No. 19	...	...	...	...	...	...	...	...	...	...	...	...
Calcine No. 20	...	...	...	...	...	...	...	...	...	...	...	...
Zircon	...	...	...	...	...	...	...	...	...	...	...	...
Maturing temperature in cones	16	15	14	14	13	15	14	14	12	12	12	13
"Te" value.	560	390	440	370	450	380	358	400	460	400	390	410

TABLE 4 (*Continued*)

47 %	48 %	49 %	50 %	55 %	63 %	70 %	72 %	73 %	74 %	77 %	78 %	79 %
60.00	50.00	50.00	51.00	50.00	50 00	50.00	37.00	37.00	....	5.00	10.00	10.00
30.00	24.00	22.00	25.00	28.50	13.50	16.40	22.00	17.60	....	....	....	....
10.00	5.00	12.00	...	...	....	....	....	....	....	....	....	....
...	1.00	1.00	1.50	1.50	1.50	1.00	....	....	....	....	....	....
...	...	...	...	...	....	....	5.00	5.00	15.00	10.00	10.00	15.00
...	20.00	15.00	...	20.00	35.00	....	....	....	....	....	....	....
...	...	...	...	...	....	4.10	....	4.40	....	....	....	....
...	...	...	22.50	...	....	28.50	....	....	....	....	....	....
...	...	...	...	...	....	....	....	....	85.00	85.00	30.00	75.00
...	...	...	...	...	....	....	36.00	36.00	....	....	....	....
10	14	14	14	14	16	14	13	13	13	13	13	13
400	410	400	390	400	540	460	500	450	640	800	790	640

TABLE 5.

Number of body.	88	94	95	107	109	116	152	180	193	194	195
	%	%	%	%	%	%	%	%	%	%	%
Kaolin.....	50.00	45.00	40.00	47.00	25.00	50.00	30.00	69.00	40.00	40.00	40.00
Feldspar.....	8.00	17.60	....	14.00	....	....	....	....	....	....	....
Potters' flint....	30.00	....	42.00	21.00	....	30.00	....	5.75	25.00	15.00	5.00
Mag. carbonate	....	....	....	10.00	....	....	....	4.58	....	....	....
Ball clay.....	....	....	....	8.00	20.00	....	10.00	9.15	10.00	10.00	10.00
Calcined kaolin	....	....	....	....	35.00	....	....	....	....	....	....
Calcine No. 8*a*	....	....	....	....	20.00	....	....	....	....	....	....
Calcine No. 13	12.00	4.40	....	....	....	20.00	....	....	....	....	....
Calcine No. 14	....	....	18.00	....	....	....	20.00	....	....	....	....
Calcine No. 19	....	....	....	....	....	....	40.00	....	....	....	....
Calcine No. 20	....	....	....	....	....	....	....	....	....	....	....
Calc. Al_2O_3....	....	33.00	....	....	....	....	....	11.52	....	....	....
Beryl.....	....	....	....	....	....	....	....	....	25.00	35.00	45.00
Maturing temperature in cones.....	15	16	20	12	16	18	16	20	12	11	11
"Te" value....	590	620	630	480	610	730	690	640	624	784	798

TABLE 6.—COMPOSITIONS OF CALCINES.

Calcine number.	$MgCO_3$. %	Kaolin %	Flint %	Calc. Al_2O_3. %	Boric acid. %	Calcination temp. (cones).
3.....	39.50	60.50	...	...	...	10
8*a*.....	14.40	44.30	41.30	...	...	12
13.....	23.85	76.15	...	...	...	12
14.....	18.20	56.00	25.80	...	...	13
19.....	...	70.20	...	27.80	2.00	20
20.....	34.30	...	65.70	...	...	16

It is at once evident from a comparison of these figures that the thermal expansion of these magnesia porcelains increases with the silica content of the body, and at the same time, is subject to decided variation within the temperature limits of 30–550° C. On the other hand, the porcelain in which the clay is lower, and all the quartz has been replaced by sillimanite, shows both the lowest thermal expansion and the least variation in the value of the coefficient. However, the composition is not the only determining factor and the importance of the micro-structure must be realized.

Summary.

The work, the results of which have been given, has been successful in showing: first, the injurious quality imparted to electrical porcelains by the use of feldspar as a flux; and second, in bringing out the desirability of replacing the quartz by minerals, or synthetically prepared materials, which are more constant in volume when heated. The remedial procedures advocated are, hence: (1) The replacement of feldspar by other fluxes, such as silicates of the type $MgO.Al_2O_3.2SiO_2$, or $MgO.Al_2O_3.4SiO_2$, or other silicates of beryllium and the alkaline earths, either natural or prepared artificially. (2) The elimination of quartz and the substitution of substances not subject to inversions or other volume changes. These may be highly calcined kaolin, alumina, zirconia or sillimanite, either natural or produced synthetically.

The compositions of the most typical porcelains produced in this work are compiled in Tables 4, 5 and 6, in which the "Te"

value is given for each body, so that these tabulations may be consulted for detailed information.

All of the electrical measurements as well as the determinations of the thermal expansions of the porcelains have been made in the Washington laboratories of the Bureau of Standards.

Bureau of Standards,
Pittsburgh, Pa.

DISCUSSION OF AN ARTICLE BY N. L. BOWEN ENTITLED "DEVITRIFICATION OF GLASS"[1] IN THE APRIL NUMBER OF THE JOURNAL OF THE AMERICAN CERAMIC SOCIETY.[1]

S. F. Cox: The able discussion of the question of devitrification of glass by N. L. Bowen recalls some work done on that subject by A. E. Williams and the writer.[2] In a study of the effect of barium salts on the physical properties of glasses, the effect of increasing barium content on the devitrification tendency was noted.

Starting with a glass of the formula $\left.\begin{matrix} 0.5\ Na_2O \\ 0.1\ ZnO \\ 0.4\ K_2O \end{matrix}\right\}$ 2.5 SiO_2, barium oxide was used to progressively replace soda, 0.1 mol. BaO replacing an equivalent amount of Na_2O, until all the latter had been eliminated; the zinc and potash were maintained constant throughout. A series would hence consist of six glasses of the same silica content. Seven such series of glasses were made, the silica content increasing by 0.5 mol. in each successive series. The last glass of the series would hence have the following composition:

$$\left.\begin{matrix} 0.5\ BaO \\ 0.1\ ZnO \\ 0.4\ K_2O \end{matrix}\right\} 5.5\ SiO_2$$

In the study of devitrification tendency, the method followed was similar to that used by Bowen. Definite heating curves were followed, the temperature being raised on successive heatings until a point well past the deformation point of most of the glasses had been attained. Fifteen of the forty-two glasses, those in the high-silica low-barium corner of the diagram, showed at 700°

[1] *J. Am. Ceram. Soc.*, **2**, 261 (1919).

[2] *Trans. Am. Ceram. Soc.*, **18**, 315–342 (1916).

C surface devitrification which was increased upon further heating at 800° C. All other glasses were entirely free from devitrification —those glasses which showed it did so only on the surface.

The tendency to devitrify at the surface of the glass is mentioned by Bowen,[1] who states that the cause is unknown. We also made note of this tendency, but were inclined to accept the explanation of Brockbank,[2] who attributes it to the volatilization of the alkali oxides from the surface, leaving a surface layer higher in silica. If the glass were nearly saturated with respect to this component, there would be a tendency for it to deposit on the surface. There would be no tendency to establish equilibrium with the glass in the interior of the piece, because of the high viscosity of the medium at these temperatures. The effect would hence be confined to the surface.

As before stated, high silica seemed more likely to produce devitrification than increasing barium content. Those glasses higher in soda, low in barium, and high in silica, tended most readily to devitrification. There was no observable connection between this property and any of the other physical properties studied.

In the light of Bowen's observations, we are inclined to believe that the reason so little devitrification was experienced in the glasses studied by us lies, among other considerations, in their high potash content and in the fact that no arsenic was used in the batches. We believe surface devitrification to have been due to the high silica content, particularly at the surface, as it occurred in those glasses highest in silica. Identification of this surface material, which was little more than a strongly adhering film, was not possible. Tendency towards crystal formation was very slow, due to the high viscosity of the glasses.

CORNING GLASS WORKS,
CORNING, N. Y.

N. L. BOWEN: Of the series of glasses mentioned by Mr. Cox, none has a content of barium disilicate as great as that of the corrected light barium mentioned on page 271. It is not surpris-

[1] *Loc. cit.*, p. 277.
[2] *Trans. Am. Ceram. Soc.*, **15**, 600 (1913).

ing, therefore, that devitrification was not noted in the barium-rich members of his series. The observance of devitrification in the silica-rich members of the series was probably due to the separation of silica, not barium disilicate.

The simple explanation of surface devitrification offered by Brockbank cannot be accepted as more than a possible contributory circumstance in some glasses. I have frequently observed that devitrification begins at the surface of glasses entirely free from alkalis or any significantly volatile constituents. A crucible full of a glass containing only CaO, MgO and SiO_2 frequently shows a surface skin of crystals of forsterite, Mg_2SiO_4.

GEOPHYSICAL LABORATORY,
CARNEGIE INSTITUTION OF WASHINGTON,
WASHINGTON, D. C.

CERAMIC ABSTRACTS.

Provisional Specifications for Glass Refractories, BY THE REFRACTORIES RESEARCH AND SPECIFICATIONS COMMITTEE OF THE COUNCIL. *J. Soc. Glass Tech.*, **3**, 3–14 (1919). *Tank Blocks:* At the request of the purchaser, the following data shall be supplied: (*a*) Analysis, giving SiO_2, Al_2O_3, Fe_2O_3, TiO_2, CaO, MgO, K_2O and Na_2O, (*b*) fusing point in cones, (*c*) porosity, (*d*) linear expansion or contraction after heating to cone 14, (*e*) crushing strength. Tentative specifications governing tank blocks follow: *Fusing point,* not under cone 30; *texture,* free from ironstone or other segregated impurity, uniformly burnt, as little black core as possible, free from holes and flaws and with true surfaces; *porosity,* shall not exceed 18% for flux-line blocks, 23% for replacement flux-line blocks, 25% for bottom-side blocks and 30% for tank-bottom blocks, the porosity being expressed as vol. of pores × 100 ÷ vol. of piece (including pores); *after-contraction* or *expansion,* test pieces about 3 × 1 to 2 × 1 to 2″ heated to cone 14 shall, when cold, show not over 2% expansion or contraction; *crushing strength,* a 4″ cube shall show at least 1,600 lbs. sq. in. *Silica bricks:* At the purchaser's request he shall be given the following: (*a*) Analysis, (*b*) fusing point, (*c*) linear expansion after heating to cone 12. Tentative specifications follow: *Analysis,* SiO_2 not under 94%, CaO not over 2%; *fusing point,* not under cone 32; *after-expansion,* not over 0.75% linear expansion after heating to cone 12; *texture,* uniform burning and absence of holes or flaws; *variations from measurements,* not over ±2%. *Silica Cement:* Shall be finely ground, contain not under 92% SiO_2 and fuse not under cone 32. *Raw Clays and Grog* are briefly covered. While manufacturers are free to use any materials and process that will give products complying with the specifications the suggestions are made as to materials and mixtures. The clay aggregate for tank blocks should usually consist of three portions: (1) *Bind clay* (a fat, aluminous clay); (2) base clay (a strong refractory clay); (3) grog (which should consist of the bind and base clays mixed together in the finely ground state, tempered, and burned to cone 14). The following compositions (after burning) and properties are suggested as indicating suitable clays. *Bind Clay:* Free silica, small proportions only; alumina, 36 to 40%; total alkalies, less than 2.5%; total fluxes, less than 4.5%. It should have a low sintering temperature but should not fuse below cone 31. *Base Clay:* Alumina, 25 to 32%; total alkalies, less than 1.4%; total fluxes, less than 4.0%; fusing point not under cone 31. The following notes are suggestions as to suitable mixtures and methods for the consideration of tank-block manufacturers: (*A*) *Tank Bottoms.* Fat plastic clay, 1 volume; lean or strong clay, 3 volumes. These clays should be well mixed in the dry, ground condition. Grog should be made by tempering a portion of the

above mixture, making it into slabs and burning to cone 10. *Mixture for blocks:* Dry, ground clay of above mixture, 5 volumes; grog $^1/_4$ to $^1/_8$" mesh, 1 volume; grog $^1/_8$ to $^1/_{16}$" mesh, 1 volume; grog $^1/_{16}$ to $^1/_{32}$" mesh, 1 volume. The mix should age for one month at least. Blocks should be slowly dried and burned to cone 10. (*B*) *Bottom-side Blocks.* Fat plastic clay, 2 volumes; lean or strong clay, 2 volumes. Grog should be made from above mixture (as for bottom blocks) and burned to cone 12. *Mixture for Blocks:* Dry ground clay of the above mixture, 5 volumes; grog $^1/_6$ to $^1/_4$" mesh, 1 vol.; grog $^1/_8$ to $^1/_{16}$" mesh, 2 vols.; grog $^1/_{16}$ to $^1/_{32}$", 1 vol. Blocks should be burned to cone 12. (*C*) *Top Side* (*Flux-line*) *Blocks.* Fat plastic clay, 2 vols.; strong clay, 1 vol.; grog should be made by burning the above mixture to cone 14. *Mixture for Blocks:* Dry ground clay of above mixture, 5 vols.; grog $^1/_8$ to $^1/_{10}$" mesh, 1 vol.; grog $^1/_{10}$ to $^1/_{32}$" mesh, 2 vols.; blocks should be burned to cone 14. (*D*) *Replacement* (*Flux-line*) *Blocks:* Fat plastic clay, 1 vol.; lower grade fat clay, 1 vol.; strong clay, 1 vol. Grog should be made by burning the above mixture to cones 6–8. *Mixture for Blocks:* Dry ground clay of above mixture, 5 vols.; grog $^1/_4$ to $^1/_8$" mesh, 2 vols.; grog $^1/_8$ to $^1/_{16}$" mesh, 2 vols.; grog $^1/_{16}$ to $^1/_{32}$" mesh, 1 vol. Blocks should be burned to cones 6–8. To avoid black-coring, the Committee recommends the use of tank blocks with no face having an area greater than 4 sq. ft. Clays suitable for pots may be classified in three grades. The lowest, or Grade 3, corresponds with the material in general use. Grades 2 and 1 are purer and more refractory. The following are limits for the composition (burned samples) and fusion temperature suggested for each grade. *Grade 1—Bind Clay.* Chemical analysis: SiO_2, 60 or less; Al_2O_3, 34 or more; Fe_2O_3, 1.8 or less; Na_2O and K_2O, 1.8 or less; MgO and CaO, 1 or less. Fusion point, not under cone 32. Mechanical Division. Clay which can be blunged and sieved to pass 80-mesh with not more than 10% residue. *Base Clay.* Chemical Analysis: SiO_2, 55 or less; Al_2O_3, 40 or more; Fe_2O_3, 1 or less; Na_2O and K_2O, 1.5 or less; MgO and CaO, 0.5 or less. Fusion point, not under cone 35. Mechanical Division. Ground to pass 80-mesh. *Grog.* Chemical Analysis: SiO_2, 50 or less; Al_2O_3, 40 or more; Fe_2O_3, 1 or less; Na_2O and K_2O, 1.5 or less; MgO and CaO, 0.5 or less. Fusion point, not under cone 35. Mechanical Division. Ground to pass 12-mesh. Temperature of firing, not lower than cone 14. *Grade 2. Bind Clay.* Chemical Analysis: SiO_2, 65 or less; Al_2O_3, 30 or less; Fe_2O_3, 2 or less; Na_2O and K_2O, 2 or less; MgO and CaO, 1 or less. Fusion point, not under cone 30. Mechanical Division. Clay which can be blunged and sieved to pass through 50-mesh with less than 10% residue. *Base Clay.* Chemical Analysis: SiO_2, 60 or less; Al_2O_3, 35 or more; Fe_2O_3, 2 or less; Na_2O and K_2O, 1.8 or less; MgO and CaO, 0.6 or less. Fusion point, not under cone 32. Mechanical Division. Ground to pass 50-mesh. *Grog.* Chemical Analysis: SiO_2, 60 or less; Al_2O_3, 35 or more; Fe_2O_3, 2 or less; Na_2O and K_2O, 1.8 or less; MgO and CaO, 0.6 or less. Fusion point, not under cone 32. Temperature of firing, not lower than cone 12. *Grade 3. Bind Clay.* Chemical Analysis: SiO_2, 70 or less; Al_2O_3, 25 or more; Fe_2O_3,

2.5 or less; Na_2O and K_2O, 2.5 or less; MgO and CaO, 1 or less. Fusion point, not under cone 28–29. Mechanical Division. Ground to 12-mesh. *Base Clay.* Chemical Analysis: SiO_2, 70 or less; Al_2O_3, 25 or more; Fe_2O_3, 2 or less; Na_2O and K_2O, 1.8 or less; MgO and CaO, 1.0 or less. Fusion point not under cone 30–31. Mechanical Division. Ground to 12-mesh. *Grog.* Chemical analysis: SiO_2, 70 or less; Al_2O_3, 25 or more; Fe_2O_3, 2 or less; Na_2O and K_2O, 1.8 or less; MgO and CaO, 1.0 or less. Fusion point, not under cone 31. Mechanical Division. Ground to pass 12-mesh. Temperature of firing not lower than cone 9. C. H. KERR.

Bottle-glass and glass-bottle manufacture. W. E. S. TURNER. *Jour. Soc. Glass Tech.*, **3**, 37–49 (1919).—Data presented in England has been largely in reference to chemical and scientific glasswares only, but the same principles hold in their application to furnace management, annealing, etc., in the bottle industry. Syphon bottles of English make are now demanded. Analysis of a typical syphon bottle glass from Bohemia follows: SiO_2, 75.34; Al_2O_3, 0.45; Fe_2O_3, 0.30; CaO, 6.83; MgO, 0.21; Na_2O, 13.78; K_2O, 3.41; Sb_2O_3, trace. This corresponds to the batch: Sand, 75.5 parts; limestone, 12.5 parts; soda ash, 27.6 parts; antimony oxide, 0.5 part; manganese (as needed). Another batch recommended for use in Germany follows: Sand, 100 parts; soda ash, 33 parts; limestone, 22 parts; saltpetre, 3 parts; saltcake, 1 part; manganese, 0.3 part; antimony oxide, 0.3 part; cobalt oxide, 0.0005 part. A similar batch recommended by the author for use in England is: Sand, 100 parts; soda ash, 33 parts; limestone, 18 parts; soda niter, 4 parts; arsenic, 0.2 part, manganese, 0.2 part. In most bottle plants the tendency to overcome troubles by liberal increases in alkali is dangerous for durability of the glass is apt to suffer. Bottles for medical and pharmaceutical preparations, also milk bottles, feeding bottles and white flint bottles generally should be tested for durability. Simple tests are (1) 1–2% morphine hydrochloride should be colorless or at most pale yellow after 24 hrs. standing; (2) 0.5–1% strychnine nitrate should show no crystals separating out in 24 hrs.; (3) 1% mercuric chloride should show no precipitation of colored oxide in 24 hrs.; (4) water with a few drops of phenolphthalein in a corked bottle should not develop pink color in 24 hrs. A better test than the above is the autoclave test carried out for 2 hrs. at 120° C. With a simple soda-lime glass the % soda ash in the batch should not exceed 28%. Of the three following batches, No. 1 yields glass that is not durable; No. 2 fairly satisfactory; No. 3 is best: Glass batch No. 1: Sand, 63.19; limestone, 7.02; soda ash, 29.78. No. 2, respectively: 63.26, 8.79, 27.94. No. 3, respectively: 62.86, 15.72, 21.42%. Production in a bottle plant hinges quite largely on the composition of the glass. General limits for easy working glasses are: Soda-lime glasses, 14% CaO; soda-magnesia glasses, 9% MgO; soda-alumina glasses, 9% Al_2O_3, these figures being percentages of the finished glass. These amounts may be increased by (1) adding other oxides; (2) reducing SiO_2. The effects of various constituents on viscosity is shown in the following summary:

Soda (*a*) makes glass more fluid, (*b*) reduces rate of setting; potash resembles soda, but is less effective weight for weight; lime (*a*) makes glass less fluid at low temperatures, but more fluid at high; (*b*) increases rate of setting; magnesia (*a*) makes glass less fluid, (*b*) increases rate of setting, but not to the same extent as lime; alumina (*a*) makes softening temperature high, (*b*) viscosity at high temperature not high compared with lime or silica; silica, causes high viscosity which does not change rapidly. A quick setting German bottle batch for use with the Owen's machine is: Sand, 1000; limestone, 554; saltcake, 300; fluorspar, 60; powdered coal, 24; manganese, 42; iron oxide, 13.

C. H. KERR.

"Silica products." M. A. BIGOT. *La Ceramique*, [1] **21**, 61 (1919).—The raw materials studied were quartzite, flint rock and sandstone. These are heated to different temps. up to 1710° C and the following properties determined: Fusibility, linear expansion, apparent density, sp. gr. by method of Le Chatelier and Candlot, absorption, microscopic examination. Fig. 1

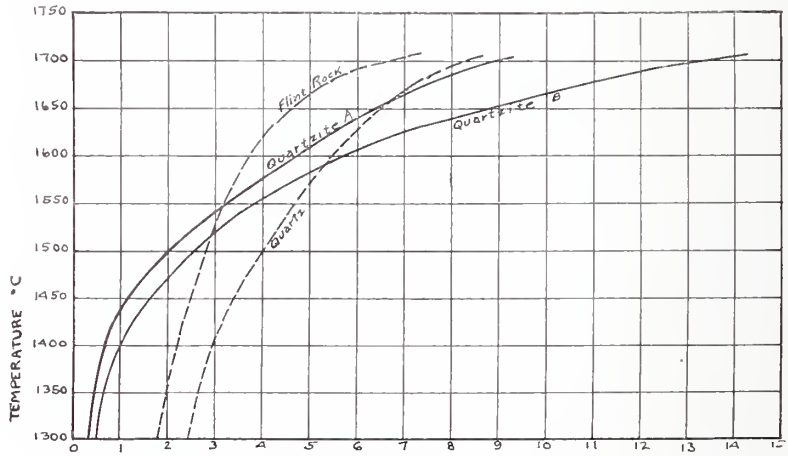


FIG 1 EXPANSION CURVES

shows the relation between linear expansion and temperature for a few silicious raw materials. Fig. 2 shows the relation between absorption and temperature. The finished products studied include those employed in the Martin or similar furnaces. These products are made of sandstone to which 2 per cent lime is added. The finished products are subjected to the above-mentioned tests and to those given in Table 1, which gives results for six commercial brick. In addition to these tests the crushing strength at different temperatures up to 1500° C were determined upon 7 different specimens of refractories by means of a special furnace constructed by Capt. Bodin. Cubes

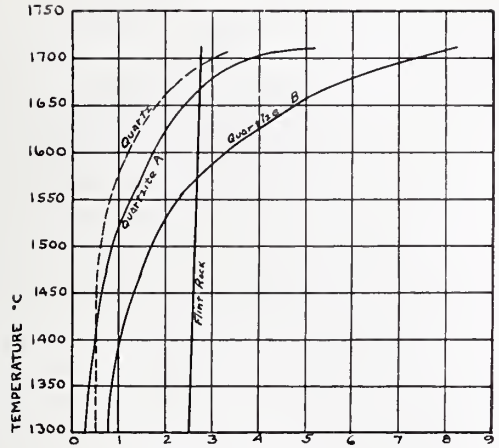


FIG 2. ABSORPTION CURVES

TABLE 1.

Designating letter	A	B	C	D	E	F
Fusion temp	1750° C	1730	1730	1730	1710	1730
Linear expansion at 1710° C.	1.60%	5.90	4.00	2.50	..	..
Absorption	12.0%	9.5	10.0	10.5	2.1	0.3
Absorption after burning to 1710° C	9.7%	16.1	12.0	9.0	..	..
Crushing strength, kg. per sq. cm. at ordinary temperatures.	121	480	160	166	1115	1415
Crushing strength, kg. per sq. cm. after burning to 1710° C.	....	0	52	..	..	..
Crushing strength, kg. per sq. cm. at 1500° C after burning to 1710° C.	77	0	0	37	52	138

A. Good commercial brick.
B. Brick which cracked at high temp.
C. Brick made of quartz rock and friable at high temperatures.
D. Brick from Martin furnace, part not attacked.
E. Brick from Martin furnace, brown part.
F. Brick from Martin furnace, gray part.

20 mm. on a side are burned to the desired temp. in a small gas-fired furnace, placed between two cylinders of refractory material and the load applied, a registering device recording the crushing load. The results are shown diagrammatically in Figs. 3 and 4 (Fig. 3 combines Figs. 8, 9, 10 and 11 in the original and Fig. 4 includes Figs. 12, 13 and 14). The reason for the high strength

of some of the refractories in the neighborhood of 1000° C cannot be stated with certainty. Edwards and Rigby[1] determined the coefficient of expansion of a refractory brick between 15° and 1180° C as 81 × 10^{-7}, and between 15° and 1180° C as 61 × 10^{-7}. This may indicate that in the neighborhood of

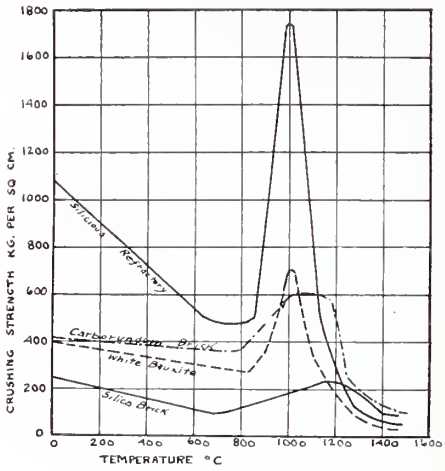


FIG 3 CRUSHING STRENGTH CURVES

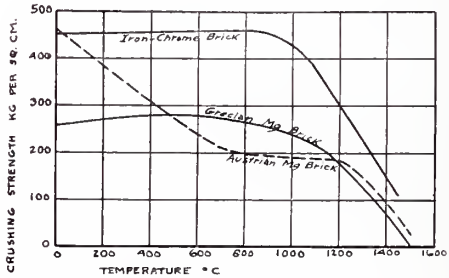


FIG. 4 CRUSHING STRENGTH CURVES

1000° C certain refractories undergo some kind of contraction which causes greater strength. If this explanation is correct, products such as magnesite and chrome brick which do not increase in strength near 1000° C have no such contraction near this temperature. F. A. KIRKPATRICK.

Summary of discussion on the setting of cements and plasters. C. H. DESCH. *Trans. Faraday Soc.*, **14**, 67 (1919).—The question as to whether the setting of a cement itself is a colloidal or a crystalline process has been studied almost exclusively in the United States and France. As regards

[1] *Trans. Eng. Ceram. Soc.*, **16**, 271 (1916–17).

the main point at issue between the supporters of the colloidal hypothesis and Le Chatelier, it is very clearly established that we are dealing largely with a difference of terms; that whether one regards the jelly as a mass of extremely minute interlacing particles or not is not of very much importance. At any rate, the essential point is that in the colloidal substance, the particles are extremely small, and therefore the surface forces are very important. With crystals of perceptible size, such as occur in the crystallization of Na_2SO_4, the surface forces are very small compared to the forces of cohesion. In the case of the ultra-microscopic particles in the colloid, the surface forces are large in proportion to the forces of cohesion. When this fact is thoroughly understood it is seen that there is no great question of fact at issue between the two views. In the setting of metals, on Rosenhain's hypothesis, we are dealing with particles very close together becoming united by exceedingly thin films. These films in the case of cements are often of greater dimensions than the particles from which they start, so that we have a mass of considerable thickness and not a mere intercrystalline film. The essential difference between cases of recrystallization and of the setting of a colloidal mass lies in the solubility. The substances which occur in cements after setting are of very low solubility. It is quite possible that the original substance present is clinker of comparatively high solubility, but that the solution formed by it is unstable and the stable products finally obtained are of extremely low solubility. F. A. KIRKPATRICK.

The mechanism of the setting process in plaster and cement. CECIL H. DESCH. *Trans. Faraday Soc.*, **14**, 1 (1919).—That part of the paper dealing with plaster of Paris and lime mortar is omitted as being already common knowledge. There are two conflicting hypotheses in regard to the setting process of Portland cement. One is the crystallization theory of Le Chatelier put forward in 1887 (see Ceramic Abstracts, THIS JOURNAL, June 1919, for a recent discussion by Le Chatelier). The alternative hypothesis was proposed by W. Michaelis in 1893.[1] On this view, while the chemical reactions assumed are those which were shown to take place by Le Chatelier, the physical conditions are supposed to be different. The hydrated metasilicate is considered to form, not a mass of radiating crystals, but a gelatinous mass or gel, the gradual dehydration of which brings about the hardening of the cement with time. The aluminate crystallizes much more readily than the silicate; but even this is regarded as forming a gel when the solution is sufficiently supersaturated, as is the case in the practical use of cement. The presence of gelatinous material has been shown by a number of workers.[2] An attempt to distinguish between the various possible colloidal products

[1] *Chem. Ztg.*, **17**, 982 (1893).

[2] Stern, *Z. anorg. Chem.*, **63**, 160 (1909); H. Ambrown, *Tonind. Ztg.*, **33**, 270 (1909); Colony, *School Mines Quart.*, **36**, 1 (1914); M. von Glasenapp, *Chem. Ztg.*, **38**, 588 (1914).

has been made by Keisermann,[1] using the method of staining with organic dyes. Others who have used this method are Wetzel[2] and Blumenthal.[3] The staining results depend largely on the concentrations and size of grain of the substances concerned and different workers find difficulty in checking each others' results. The U. S. Bureau of Standards (Tech Papers Nos. 43 and 78) has successfully used the staining method and has been able thus to identify the various gelatinous materials. The real nature of the colloidal condition has been unnecessarily obscured by the barbarous terminology which has been adopted by so many writers on the chemistry of colloids, and especially by the school of Wo. Ostwald. Stripped of these uncouth technical terms, the study of colloids appears far less formidable and it is hoped that the putting in relief of the view of the two rival hypotheses as to the setting and hardening of cement may lead to a recognition of the true facts of the case, and to an elimination from the subject of the complications which owe their origin largely to a defective terminology. F. A. KIRKPATRICK.

Discussion on the setting of cements and plasters, WALTER ROSENHAIN. *Trans. Faraday Soc.*, **14**, 52 (1919).—The setting of cement appears to be one of a whole class of phenomena of an analogous character, and this analogy may be extended still further to include the setting of metals when they solidify from fusion. The latter process is like the former because we see in it the formation of an aggregate of coherent crystals instead of merely a heap of loose crystals lying one upon another, and an aggregate of crystals adhering powerfully to one another is very much what we have in a plaster or cement after setting. We can dismiss at once as the cause of cohesion the idea of mere frictional adhesion due to any sort of interlocking. We have cohesion due to that cause in textile fibres where the frictional adhesion between intertwined units may become very large compared with the strength of any one unit. The hypothesis now generally accepted as to the cause of the adhesion of crystals in metals is that a thin film of amorphous material acts as a binder between the crystals. We may regard cement as consisting, at one stage, of solid grains or crystals lying surrounded by a liquid solution of certain constituents. This liquid then undergoes solidification as the result of the removal of water—either by percolation or by chemical action, such as the hydration of some of the solid material. If the film of liquid is thick enough, this solidification may take place by crystallization, but if it is very thin it may—by analogy with the phenomena in metals—congeal in an amorphous condition. Even if there is crystallization, the final thin films of liquid between the crystals will still solidify as "undercooled," or supersaturated liquid. It is these amorphous layers which give to the whole mass its great stiffness and strength. This theory, which has proved useful in explaining

[1] "Der Portlandzement, sieve Hydratbildung and Konstitution," Dresden (1910).

[2] *Zentr. Hydrauk. Zemente*, **2**, 34 (1911).

[3] Dissert., Jena, 1912; *Silikat Z.*, **2**, 43 (1919).

the setting of metals, should aid in the analogous phenomena met with in the setting of cement. F. A. KIRKPATRICK.

Discussion on setting of cements and plasters. E. HATSCHEK. *Trans. Faraday Soc.*, 14, 53 (1919).—The reason for the different results obtained by Le Chatelier and Michaelis in regard to the setting of cement is that one worked in dilute and the other in concentrated solution, and this is practically the whole difference, and accounts for their divergent results and conclusions It is well known, especially from the work of von Weimarn on about 200 different substances, that slightly soluble compounds may be obtained either as gels, as the familiar crystalline precipitation, or as sols, simply by an appropriate choice of the concentrations of the reacting solutions. Thus, $BaSO_4$ may be obtained as a clear gel by using concentrated solutions of two exceptionally soluble salts, namely BaSCN and $MnSO_4$. The two theories are not only compatible but even complementary. It is perfectly possible that the first product of a reaction may be a gel consisting of ultra-microcrystalline elements and that this may gradually assume a coarser crystalline structure owing to the intermediate true solution of the reaction product. One case has been described by W. Doehle,[1] who obtained the Hg salt of a sulphonic acid which formed gels in concentrations as low as 1 : 300. The gel gradually breaks up and the salt crystallizes out, the process being irreversible. Another, even more striking case, is that of camphoryl phenyl thio-semicarbazide studied by M. O. Forster.[2] This substance has a true solubility of about 0.5 per cent in cold abs. alc., but is very sol. in boiling alcohol. If the latter solution is allowed to cool slowly, the substance crystallizes out in large, simple crystals. If, however, the solution is cooled suddenly, it sets to a beautiful gel, recalling silicic acid gel. This gel is not stable but in a short time—hours, or at most, days, depending on temperature and concentration—the gel exudes liquid, while at the same time the substance crystallizes out. The crystals thus formed are all twins or more complicated aggregates. This is an instance in which gel formation and subsequent crystallization succeed each other with striking rapidity, owing to the high true solubility of the substance in the cold solvent. Intermediate cases between this and the transformations requiring years or possibly centuries, can, no doubt, be found by a careful study of solubilities.

F. A. KIRKPATRICK.

CERAMIC PATENTS.

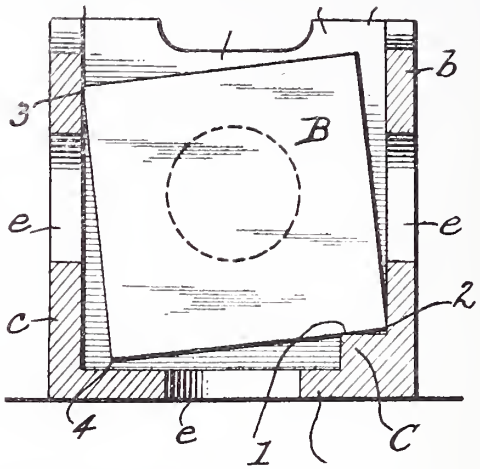
GEORGE E. MIDDLETON, *Abstractor.*

Sagger. H. D. LILLIBRIDGE. U. S. 1,302,684, May 6, 1919. This invention relates to a saggar used in burning floor or wall tiles. Because of the

[1] Ueber Derivate des Benzothiazols, Dissertation, Leipzig, pp. 14, 61 (1912).

[2] *Kolloid Z.*, 11, 158 (1912).

longitudinal shoulder C at one side of the base of the saggar, the tiles are supported from three points, 1, 2 and 3, the lower corner 4 being unsupported



as shown (see Fig.). The frictional resistance offered by the three points of support prevents displacement of the tiles.

Brick-cutting machine. G. F. Pemberton. U. S. 1,302,964. May 6, 1919. A machine for cutting freshly-made, blank, plastic blocks into irregular shapes for use in arches and elsewhere where tapering or trapezoidal shapes are required. The cutter comprises a frame across which wires are strung at the desired angles. The blank is placed on a base plate and this cutting frame forced down through it.

Glass and method of making the same. B. F. Drakenfeld, Jr. U. S. 1,303,268, May 13, 1919. Amblygonite, a lithium-aluminium-fluophosphate of a chemical formula approximating $Li(ALF)PO_4$ is added to a glass batch, the amblygonite being in a fused condition. When working with a basic batch to be used in the manufacture of jars, bottles, window, plate or other inexpensive glass, from 1 to 5% is added. When making opaque or translucent glass the addition of amblygonite is up to 50% of the weight of the batch. The fluorine and phosphate contents assist in imparting opacity. In all cases the strength, temper and appearance of the glass are said to be improved. Vitreous enamels and glazes are included as well as glasses.

Glass and method of making the same. B. F. Drakenfeld, Jr. U. S. 1,303,269, May 13, 1919. Spodumene, a lithium-aluminium-metasilicate of a chemical formula approximating $LiAl(SiO_3)$ is added to a glass batch. It is said to reduce the coefficient of expansion of the glass. The properties of the resulting glass and the methods of procedure are similar to those discussed under patent 1,303,268, above.

Brick machine. H. R. McCune. U. S. 1,303,344, May 13, 1919. This invention relates to means for roughening and ornamenting the ends and one face of stiff-mud bricks. As the clay ribbon issues from the die of the brick-machine, it is first passed between two brushes rotating vertically in a direction counter to the motion of the clay, these brushes roughening the sides of the clay ribbon, and then under a third brush horizontally rotating in a direction counter to that of the clay, this brush roughening the top of the ribbon.

Brick conveyor. B. S. Moore. U. S. 1,303,359, May 13, 1919. A mechanism for conveying and handling brick, especially adapted for the receiving of the bricks as they are delivered from the molding machine, carrying the same in bulk to another point and depositing them on cars in suitably arranged layers for future handling.

Process of and apparatus for drawing glass. H. T. Bellamy and J. C. Smith. U. S. 1,303,452, May 13, 1919. This invention relates to the drawing of thin-walled glass articles, the specific article illustrated being a cylindrical bulb for small incandescent lamps.

Clay-mixing machine. H. R. Straight. U. S. 1,303,611, May 13, 1919. The mixer consists of an endless conveyor, curved upward to form a trough. Power is applied so that the lower run is taut and the upper being very loose and supported upon a plurality of transverse rollers, presents an undulating surface. Crushed clay is fed onto this trough-shaped conveyor at one end, together with the required water. The mixture then passes under a horizontally rotating cylinder having outstanding pins which act as mixers and then along the undulating surface of the conveyor to the discharge end.

Continuous kiln. D. R. Bone. U. S. 1,303,760, May 13, 1919. This is a continuous kiln of the ring type employing temporary combustible partitions between the green and preheating ware. Separate, auxiliary furnaces are provided for water smoking. Powdered coal for the burn is fed through the platting by means of a rotary feeder which is designed to exclude the entry of air. The inventor seeks to avoid the admission of more air than is necessary completely to burn the fuel used.

Refractory. F. J. Tone. U. S. 1,303,993, May 20, 1919. This invention relates to refractory articles such as crucibles. The composition consists of graphite 40 parts, sintered magnesia 40 parts, plastic clay 20 parts. Such a mixture is said to be easy to mold and work in the plastic state and after burning has a body which is very refractory, less oxidizable than graphite, of superior strength, of high conductivity and great durability.

Manufacture of Keene's cement. J. C. Best. U. S. 1,304,148, May 20, 1919. A process of manufacturing Keene's cement which consists in burning gypsum in a rotary internally-fired kiln for a period of substantially one hour at a temperature of from 1200° to 1400° F., admixing with the burnt product one per cent of a neutral catalyst such as K_2SO_4 and grinding to 150 mesh. This cement is particularly adapted for making artificial marble or scagliola.

Shaft kiln. E. SCHAMATOLLA. U. S. 1,304,514, May 20, 1919. A vertical shaft-kiln for burning lime, magnesite and other rock products with producer gas. The object is to prevent overburning of the product near the lining and gas inlets and underburning near the center, or down-rolling of unburnt rock through the center. This is accomplished, first, by the use of two series of draw openings, the one for the material near the wall and the other for that in the center, by means of which the product near the walls can be drawn oftener than that in the center, and second, by allowing a part of the air needed for combustion to enter at the base of the column whereby complete combustion and overburning near the gas inlets is obviated.

Copies of the above patents may be obtained for five cents each by addressing the "Commissioner of Patents, Washington, D. C.

ACTIVITIES OF THE SOCIETY.

The following motions have recently been passed by the Board of Trustees:

July 8, 1919. It was voted that all papers presented before the Sections and Divisions of the Society be subject to prior publication in the Journal, and that this be incorporated in the Rules of the Society.

July 15, 1919. It was voted to loan a set of the Transactions of the Society, including the JOURNAL, to the Division of Chemistry and Chemical Technology of the National Research Council, as part of a technical library.

July 15, 1919. Credentials were issued to Dr. E. W. Washburn as a delegate to the International Chemical Council, called together in London, for the purpose of completing the organization of an International Chemical Union.

NECROLOGY.

Clarence W. Hathaway, metallurgist for the past eight years for the Granite City Steel Works Branch of the National Enameling and Stamping Co., met his death by drowning while swimming with a party of friends in the Mississippi River, on July 19, 1919, at Granite City, Illinois.

He was born in Columbus, Ohio, in 1886, and was educated in the schools of that place and at the Ohio State University. He had previouly been employed with the U. S. Steel Corporation at Gary, Indiana, before accepting his last position. He was a man of exceptional ability, a keen investigator and a tireless worker.

Mr. Hathaway was a member of the Masons and Elks, American Chemical Society, Metallurgical Society of America, and the American Ceramic Society. He leaves a wife, father and mother, one brother and four sisters.

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EDITORIALS.

CERAMIC DAY AT THE CHICAGO EXPOSITION.

The Fifth National Exposition of Chemical Industries will be held at the Coliseum in Chicago the week of Sept. 22, 1919. The Exposition has become a national institution and each year sees a wonderful development in the display of chemical products and appliances and in the interest taken by the ever-increasing number of those in attendance.

Expositions in Europe are of far greater industrial significance than in the United States, but we are rapidly learning the great good to be accomplished by such an exchange of ideas and data as can be accomplished only by expositions. President McKinley once said, "Expositions are the timekeepers of progress" and we are beginning to realize the clearness and correctness of his view.

The management of the Fifth National Exposition has set aside Wednesday, Sept. 24th, as Ceramic Day and a special effort will be made on all sides to make that day of unusual interest and value to all who are interested in any branch of the silicate industries. Scattered throughout the hall will be the individual exhibits of the many companies, displaying the latest developments in the various lines of structural materials, refractories, glass, white ware, porcelain, enamels, abrasives, and the many other branches allied with the ceramic family.

Special interest will center in the ceramic program of the day. Many interesting things are promised including the following addresses and "movie" films:

Afternoon—Sept. 24th:

"Superior Refractories," by Mr. Ross C. Purdy, Norton Company, Worcester, Mass.

"Buy on Analysis," by Dr. Alexander Silverman, University of Pittsburgh, Pittsburgh, Pa.

Evening—Sept. 24th:

Film—Making of Cut Glass.

"The American Ceramic Society—Past, Present and Future," by Prof. Charles F. Binns, Alfred University, Alfred, N. Y.

Film—Manufacture of Glass.

"The Application of Scientific Methods to Ceramic Research," by Mr. A. V. Bleininger, Bureau of Standards, Pittsburgh, Pa.

This will serve to give an idea of the excellence of the program that awaits those who meet with us in Chicago. Other very interesting contributions are promised and the final program will be one of first importance to those interested in the science of the silicates.

The headquarters of the American Ceramic Society will be at Booth No. 205 in the Balcony of the Coliseum. Representatives of the Society will be in constant attendance at the Booth throughout the Exposition to assist visitors in any possible way. Make your headquarters at Booth 205 with the other ceramic men and take advantage of the facilities provided by the American Ceramic Society for your comfort and convenience.

The Chicago Section of the American Ceramic Society is making full preparations to look after the Society's interests and in all probability a dinner will be arranged at which we can all get together between the afternoon and evening sessions on Wednesday. Closer association and closer contact between the various individual interests and the various interests included in the silicate industries are the very great needs of our very large and growing family.

Let us all meet in Chicago on Wednesday, Sept. 24th.

C. H. Kerr, *Chairman*, Committee on Coöperation.

NEW MEMBERS.

With the approach of Fall and cooler weather the activities of the various committees of the Society should be pursued with

new energy. The vacation period is always one of marking time and more or less disorganization. The steady upward trend of the membership-growth curve of the Society should be maintained. The increase of more than 100 per cent in membership within the past two years has been very gratifying and indicates an awakening to the necessity of the technical advancement of the industry through the coöperation of all branches of the industry.

It is imperative that each and every member of the Society lend his utmost support to the newly appointed chairman of the Membership Committee, Mr. F. H. Riddle, in his campaign for new members. After all, personal solicitation is the best means of recruiting members and unless the efforts of the Chairman are backed up by our members his efforts are apt to prove discouraging.

The large acquisition of members from the glass industry during the past two years has been very gratifying. However, this great branch of the ceramic industry has been scarcely touched and should prove an ever-increasing bulwark to the Society. There are other fields which, if thoroughly canvassed, should prove fertile sources for new members. Considerable space has been devoted in the Transactions and in the Journal to discussions of the technique of cement and the lime industries and it is to be hoped that these two branches of the ceramic industry will lend their increasing support to the American Ceramic Society. The designation of the Journal as the official technical organ of the National Lime Manufacturers' Association was a long step forward in securing the coöperation of these great industries in the work of the Society. An active campaign for recruiting members from the individuals and firms engaged in this industry should not be neglected.

Boost for new members!

ORIGINAL PAPERS AND DISCUSSIONS

ON THE EFFECT OF EXTRACTION UPON THE PLASTICITY OF CLAY.

By W. A. Hamor and H. E. Gill.[1]

An examination of the literature relating to clays shows that very little experimental work has been conducted for the purpose of ascertaining the effect of organic solvents upon that property of clay known as plasticity. Statements have been repeatedly made that the plastic properties of clays are influenced by their content of organic matter, but there has been an insignificant amount of research evidence presented, either in opposition to or in support of this theory.

The report of Otto Nolte[2] may be summarized as follows:

A heavy Silesian clay was thoroughly extracted with ethyl ether and acetone. On evaporation, a yellow mass remained which consisted of sulphur and an unidentified organic substance. Extraction did not diminish the plasticity of the clay and Nolte was unable to verify Böttcher's conclusion that this property is predominately influenced by the organic content.

The effect of organic matter upon plasticity was brought out by Böttcher. The same action was noted by E. Bourry,[3] who indicated the sources of organic matter in clay and remarked that the proportion of organic substances is not usually large but, generally speaking, tends to increase the plasticity and water-absorbing power. W. B. Davis[4] has more recently discussed the rôle

[1] The authors are indebted to Dr. E. W. Tillotson, Assistant Director of the Mellon Institute, for his courteous and suggestive interest.

[2] *Biedermann's Zentr.*, **47**, 108–9 (1918).

[3] "A Treatise on the Ceramic Industries," 28 (1911).

[4] *Trans. Am. Ceram. Soc.*, **16**, 65 (1914).

played by organic substances. Davis states that "clays are simply buried soils, or soils reworked by water, so we may expect to find these organic colloids present to some extent. Whether they are present as emulsion colloids, and acting as protective colloids, has yet to be definitely determined. We do know that many of the excessively plastic clays often contain considerable organic matter. These organic colloids and adsorbed salts tend to raise the viscosity of the water-film, hence increasing the plasticity." In view of these and other similar statements in the literature, and because of the absence of data on American clays, the authors carried out the extraction of several widely distributed samples, not with the purpose of supporting any one theory, but solely to determine the deportment of American clays after the removal of soluble organic material.

In the extractions two varieties of clays were used, namely, ball clays and fire clays. These were taken from widely separate fields, those of Mississippi, Missouri and Kentucky. A brief description and the analysis of each clay upon which the extractions were made are as follows:

First.—A white colored, fairly plastic, fire clay from the Missouri district; it is used in glass-house refractories, high-grade brick, and graphite crucibles.

Analysis.

	Per cent.
SiO_2	50.29
Al_2O_3	32.58
Fe_2O_3	0.92
MgO	1.11
CaO	1.00
TiO_2	1.44
Alkalies	1.86
Loss on ignition	10.44

Second.—A Kentucky ball clay, which possessed a brown color, very high plasticity, and exhibited cracking qualities when made into briquettes and dried. An enumeration of the uses of this type of clay would include those already mentioned, namely, glass pots and crucibles; abrasive wheels, saggers, and lead pencils may be added.

Analysis.

	Per cent.
SiO_2	51.40
Al_2O_3	33.78
CaO	1.04
Fe_2O_3	1.39
MgO	0.81
TiO_2	1.17
Alkalies	0.94
Loss on ignition	10.11

Third.—A Mississippi ball clay of the Tallahatchie County deposit, described as tough, of high plasticity, dense-burning nature, and often possessing high transverse strength. The clay of this deposit finds extensive use in the crucible and glass-pot industries and frequently as a bond in the manufacture of abrasive wheels. An analysis of the clay of the Mississippi district has not been carried out, but it has been reported to be high in silica and the iron oxide is said to run from 1.5 to 4.5 per cent.

Experimental.

The clays were ground to 80-mesh and dried over night at a temperature of 110° C. After being cooled to room temperature, a 450-gram sample was weighed out and transferred to a narrow-neck glass-stoppered bottle of $1^1/_2$ liters capacity. The next step was the addition of the solvent, exactly 500 cc. being added to each sample of clay. Care was taken to use the same amount of solvent in each experiment, so that the resulting action of each solvent on the various samples of the same clay might be comparable. After the organic solvent had been added, the bottles were each in turn placed in a shaking machine and agitated for 14 hours. Upon the expiration of this time the mass was removed immediately from the extractor and filtered. The residue was dried at 110° C over night and again passed through an 80-mesh sieve. This latter precaution, passing the clay for a second time through a sieve, was taken to make certain that the clays were in a uniform state of division, so that the change in plasticity, if any resulted, might not be due to a variation in the size of the particles.[1]

[1] The authors desire to express their appreciation of the helpful suggestions received from Mr. M. G. Babcock, an Industrial Fellow of the Mellon Institute, during the progress of this experimental work.

The organic solvents used were ethyl alcohol (95%), ethyl alcohol containing 1 per cent of HCl, commercial benzol, acetone and carbon tetrachloride. The filtrates of each were received in separate containers and were evaporated to 50 cc. on a water-bath for examination. In each case, however, the quantity of extracted matter was too small for the identification of the constituents.

Methods Used for the Determination of Plasticity.

First.—The Method of A. V. Bleininger and G. A. Loomis,[1] one of the most reliable procedures, was used as follows to determine the relative plasticities:

Water of Plasticity.—The samples were separately mixed to the point where their water contents gave to the clay a softness and pliability such that the samples could be easily worked without adhering to the hands. This was taken as the point of greatest workability. The clays were rolled into cylindrical shapes and wrapped separately in wet cloths, after which they were placed in tightly capped jars for three days. The moisture being uniformly distributed at the end of this period, each sample was ready for molding. They were each thoroughly worked and molded into briquettes of the dimensions $1'' \times 2\frac{1}{2}'' \times \frac{1}{2}''$. The weight of each briquette was determined and recorded as the wet weight. After drying at room temperature and then to constant weight at 110° C, the dry weights were determined and the water of plasticity was calculated as the ratio of the loss in weight during the drying process over the dry weight. Three determinations were made on each sample and all results recorded in the following table are the average of three similar determinations and not of one chance result.

Drying Shrinkage.—The shrinkage was determined by weighing the wet briquettes, then immediately suspending them in kerosene and again making a suspended reading. This gave the wet volume. The specimens were dried, first at atmospheric temperature, and later to 110° C. The dry weights were then determined. The briquettes were immersed in kerosene for twelve hours and the

[1] *Trans. Am. Ceram. Soc.*, **19**, 601 (1917).

dry volumes determined as before. From these determinations the data presented in Table 1 were obtained, a brief explanation of which may be given as follows:

$$a = \left\{ \begin{array}{l} \text{Volume of total water in} \\ \text{terms of true clay volume,} \\ \text{expressed in per cent.} \end{array} \right\} = \frac{100\ S\ (W - D)}{D}$$

recorded in first column, Table 1.

$$b = \left\{ \begin{array}{l} \text{Volume of shrinkage water, in} \\ \text{terms of true clay substance,} \\ \text{expressed in per cent.} \end{array} \right\} = \frac{100\ S\ (V - V_1)}{D}$$

recorded in second column, Table 1.

In the computations for which the above formulas are given:

S = specific gravity of clay, which approaches 2.60 for these types of clays.
D = dry weight of sample.
W = wet weight of sample.
V = wet volume of sample.
V_1 = dry volume of sample.
(a — b) = pore water in terms of true clay volume.
$\frac{(a - b)}{b}$ = the relation of pore to shrinkage water.
$\frac{b}{a} \times 100$ = per cent of shrinkage water in terms of the volume of total water.

Second.—The Atterberg Method[1] was also used as follows to determine the relative plasticities:

(*a*) *The Flow or Upper Boundary of the Plastic Condition.*—About five grams of the clay powder of approximately 120-mesh were put into a small porcelain evaporating dish and made into a paste by the addition of distilled water. By means of a polished nickel spatula the mass was shaped into a smooth layer, a trifle less than 1 cm. (0.39 in.) in thickness. The clay was divided into two portions by cutting a triangular-shaped channel through the mass, the lower edges being separated a trifle. The dish was then repeatedly and sharply rapped against the heel of the hand in order to bring about the flowing together of the separated portions. The flow limit was reached when the two portions of the clay could barely be made to meet at the bottom. If the paste was too thick or too thin, water or clay was added

[1] Bur. Standards Tech. Paper No. 46 (1915).

TABLE 1.—EXPERIMENTAL RESULTS, METHOD NO. 1.

Type of clay.	Solvent used in extraction.	% of H_2O of plasticity $\frac{W-D}{D} \times 100$.	H_2O in terms of clay (true) volume (a).	Shrinkage H_2O in terms of true clay volume (b).	Pore H_2O in terms of true clay volume (a—b).	Ratio of % pore to % shrinkage H_2O $\frac{a-b}{b}$.	Shrinkage H_2O in terms of total H_2O $\frac{b}{a} \times 100$.	% volume shrinkage in terms of dry volume $\frac{v-v_1}{v} \times 100$.	Wet volume of samples in cc. av. of three V.	Dry volume of samples in cc. av. of three V_1.
Missouri fire clay	none	31.7	82.4	39.93	42.47	1.063	48.46	27.9	20.79	16.25
Missouri fire clay	aeetone	28.0	72.8	33.17	39.63	1.195	45.56	23.8	19.82	16.01
Missouri fire clay	aleohol + 1% HCl	26.1	68.0	28.40	39.60	1.394	41.76	20.4	20.00	16.61
Missouri fire elay	aleohol	25.3	65.8	30.48	35.32	1.159	46.32	22.5	21.14	17.24
Missouri fire elay	benzol	23.1	60.1	26.30	33.80	1.285	43.76	19.6	21.42	19.90
Missouri fire elay	earbon tetraehloride	21.0	54.6	18.53	36.07	1.946	33.93	13.3	19.74	17.41
Kentucky ball elay	none	71.8	186.7	144.8	41.9	0.2894	77.56	99.20	22.28	11.18
Kentueky ball elay	benzol	66.6	173.6	131.2	42.4	0.3300	75.57	90.26	22.17	11.65
Kentueky ball elay	aleohol	65.3	169.8	130.6	39.2	0.3002	76.93	90.23	20.39	10.72
Mississippi elay	none	56.87	147.8	99.82	48.04	0.4849	67.54	65.35	20.07	12.14
Mississippi clay	aleohol	51.78	134.6	88.68	45.95	0.5181	65.93	59.99	21.50	13.44

until the proper consistency had been attained. The water content of the clay mass in this condition was then carefully determined by drying to constant weight and the value expressed in terms of percentage of the dry weight of the clay.

(*b*) *The Lower Plasticity Limit or the So-called Rolling Limit.*—Clay in the stiff plastic condition was rolled between the hands and a plain smooth surface covered with paper until slender cylindrical threads formed. These "threads" might break up into smaller lengths, but this was of no significance. Dry clay powder was added to the plastic mass and thoroughly incorporated in it, and the rolling operation repeated. The desired consistency had been attained when the clay could no longer be fashioned into threads by this method, but crumbled instead. This condition could be recognized rather sharply, and no difficulty was experienced in checking the results. The water was determined from this consistency and expressed in terms of percentage of

TABLE 2.—EXPERIMENTAL RESULTS, METHOD NO. 2.

Clay.	Solvent.	Av. upper limit.	Av. lower limit.	Plasticity number.
Missouri fire clay	none	31.31	22.76	8.55
Missouri fire clay	alcohol + 1% HCl	26.47	18.23	8.24
Missouri fire clay	alcohol	25.04	17.22	7.82
Missouri fire clay	acetone	28.21	20.74	7.47
Missouri fire clay	carbon-tetrachloride	28.41	21.78	6.63
Missouri fire clay	benzol	25.59	19.26	6.33
Kentucky ball clay	none	68.91	36.09	32.82
Kentucky ball clay	benzol	63.65	32.00	31.65
Kentucky ball clay	alcohol	63.87	33.07	30.80
Mississippi ball clay	none	51.16	25.23	25.93
Mississippi ball clay	alcohol	52.53	28.84	23.69

the dry weight of the clay. This figure subtracted from the one determined by (*a*) gives a figure which was called the "plasticity number" and the higher this value the more plastic is the clay.

By this procedure, as by the first, several determinations were made and the averages reported as shown in Table 2.

Discussion of Results.

It will be noticed, under the title column, "Solvents used in Extraction," that in each table three are marked "none." These determinations were made on the clay as it was received with no previous extraction and from these the actual plasticity of each was accurately determined. With these results the plasticity of each extracted sample was compared with that of the unextracted sample of its type.

The results show that in each extraction there was a diminution which was established by one method and checked by the other. It will be seen from the tables that the two determinations agree rather closely. While the extent of change does not agree by both methods, the results are comparable. Upon the Missouri clay the three solvents having greatest effect were alcohol, alcohol containing 1 per cent of HCl, and acetone. These three are grouped together in both determinations; in Table 1 the acetone extraction stands first as having the least effect, while in Table 2 it is third, with alcohol and alcohol containing 1 per cent of HCl retaining their same relation to each other. The carbon tetrachloride extraction and that of benzol check very closely, notwithstanding that they alter places with each other.

In the results on the Kentucky ball clay it will be seen that they retain the same relation to the unextracted sample by both the Bleininger and Loomis and the Atterberg methods.

Summary.

The results obtained may be summarized as follows:

1. Certain clays after being treated with organic solvents are less plastic than the original materials.

2. The degree of lowering of the plasticity is not the same for different solvents.

Mellon Institute of Industrial Research,
Pittsburgh, Pa.
July 10, 1919.

A MACHINE FOR TESTING THE HOT-CRUSHING STRENGTH OF FIREBRICKS.[1]

By H. G. Schurecht.

Introduction.

In testing refractories under load, Bleininger and Brown[2] applied a constant load of 25–50 pounds per square inch on firebricks and after heating to a constant temperature (1150–1500° C) measured the deformation.

Mellor and Moore[3] tested fire-clay brick by applying a constant load and increasing the temperature until the brick deformed a fixed amount and used this "squatting" temperature as a means of comparing fire clays.

Shaw[4] used a compressed spring to apply the load—in which case the load increased as the brick expanded and decreased as the brick contracted—his claim being that this test brought the conditions closer to those encountered in practice.

After freely consulting the works of these authors a machine was designed by the Bureau of Mines with which the hot-crushing strength can be measured in addition to the deformation under a constant load. The strength test is of value in differentiating between silica, magnesite, chrome and high-grade fire-clay bricks when heated from 1300–1500° C. It is also of value for testing lower-grade refractories at low temperatures; for testing sagger bodies; and for measuring the strength of fire-proofing materials at various temperatures.

Description of Testing Machine.

The furnace (Figs. 1 and 2) is fired by two tangential air-gas burners. It is capable of testing two bricks at the same time and can be controlled by one operator.

[1] By permission of the Director, U. S. Bureau of Mines.

[2] A. V. Bleininger, and G. H. Brown, *Trans. Am. Ceram. Soc.*, **12**, 337–369 (1910); Report of Committee C-8 on Refractories, *Proc. Am. Soc. Testing Materials* (1918).

[3] J. W. Mellor, and B. J. Moore, *Trans. Eng. Ceram. Soc.*, **152**, 117–130 (1915–1916).

[4] J. B. Shaw, *Trans. Am. Ceram. Soc.* **19**, 498–506 (1917).

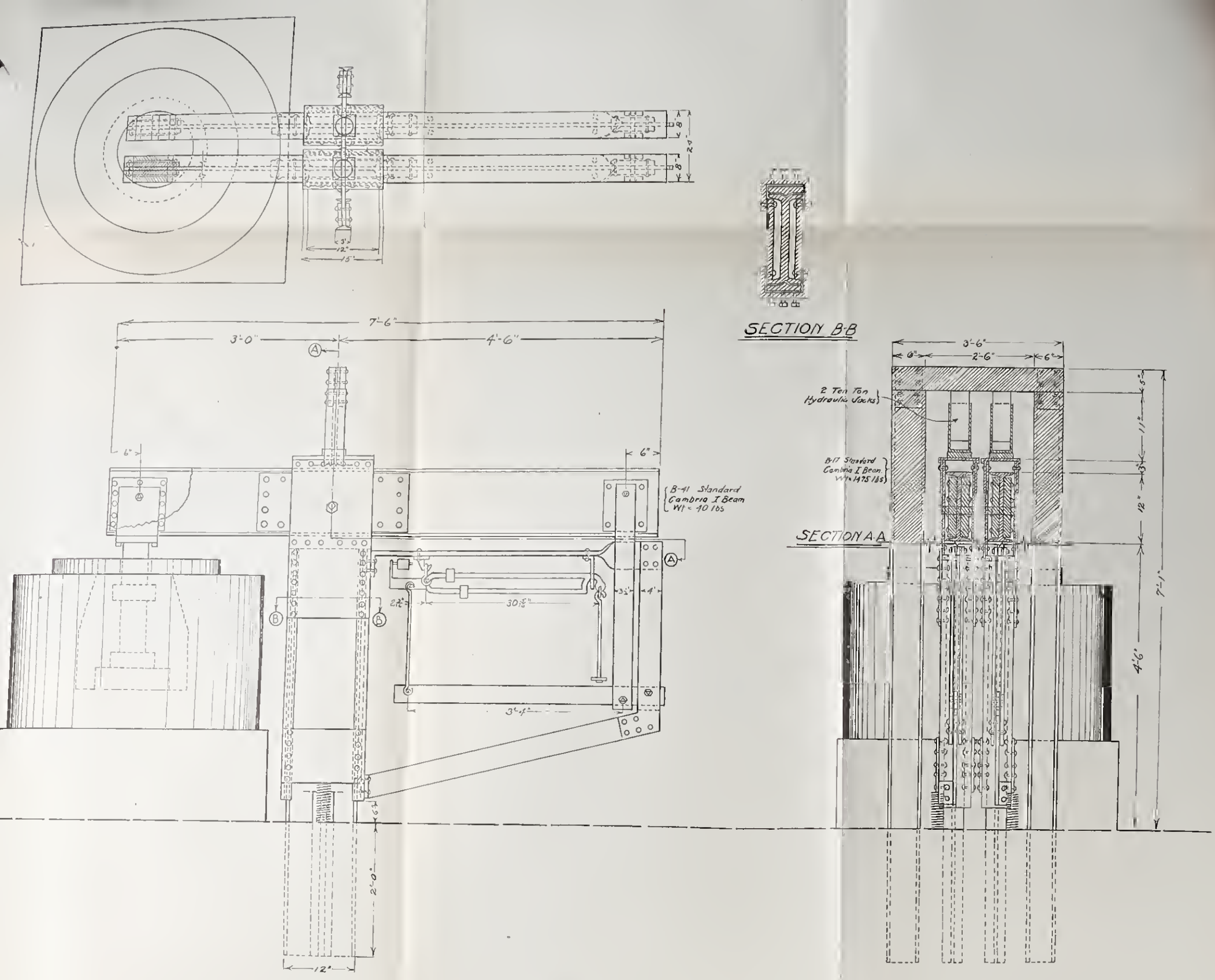
7'-6"
3'-0"
4'-6"
6"
6"
B-41 Standard
Cambria I Beam
Wt = 40 lbs
30⅝"
3'-4"
2'-0"
12"
SECTION B-B
3'-6"
6"
2'-6"
6"
2 Ten Ton
Hydraulic Jacks
B-17 Standard
Cambria I Beam
Wt = 14.75 lbs
SECTION A-A
4'-6"

FIG. 1

In testing a fire-clay brick at temperatures above 1200° C, the brick deforms considerably before the final failure occurs. It was therefore necessary to design a machine that would take care of this deformation without tilting the main test-beam—as this would cause shearing of the brick and a premature failure. This is accomplished by moving the whole testing machine down

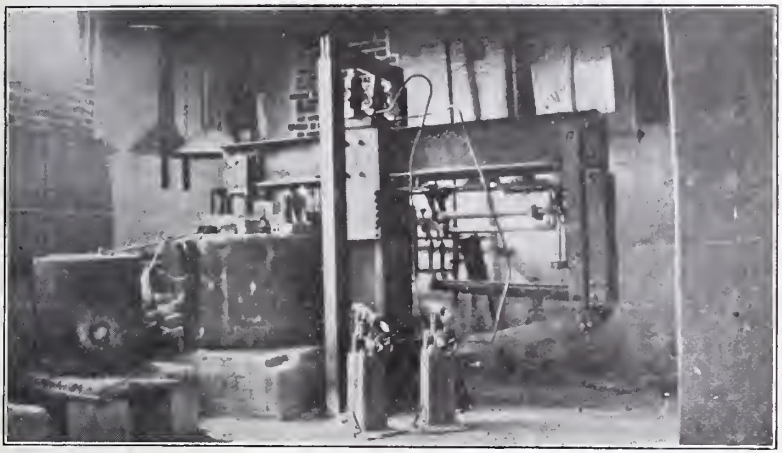

FIG. 2.

as the brick deforms. Each machine operates independently of the other and both are guided in a straight up-and-down movement by means of four slides which fit in V-shaped lateral grooves in stationary vertical I-beams. When no pressure is applied, the machine is supported by means of springs (see Fig. 1).

Method of Testing Brick.

The brick are subjected to a load of 25 pounds per square inch while the temperature is being increased to the desired point. The final temperature is varied in testing different materials as follows:

For fire-clay brick	1300° C
For magnesite brick	1350° C
For chrome brick	1350° C
For silica brick	1500° C

In each case the heating curves recommended by the American Society for Testing Materials[1] are used. At the end of the heating, the load is increased by applying pressure by means of two Watson-Stillman hydraulic jacks until the brick fails—the load then being measured by keeping the scale-beams balanced. The use of an automatic scale in the place of the scale-beam makes the measurement of the load much simpler.

The machine is accurate to 5 pounds per square inch—an accuracy of 1.6 per cent for minimum loads of 25 pounds per square inch on a standard brick, or 0.2 per cent for a load of 200 pounds per square inch (which is about the average pressure used). The safe maximum load for each machine is 8000 pounds.

By means of an Ames dial placed against the main testing beam, the expansion and contraction of the brick may be measured throughout the test. Corrections are made for the expansion of the plungers but the expansion thus recorded is only approximate, owing to the expansion of the furnace bottom.

Results of Tests.

The results of tests are shown in Figs. 3, 4, 5, 6 and 7. The silica brick marked "Ohio" was made of flint from near Flint Ridge, Ohio, while the one marked "Pennsylvania" was made from a Pennsylvania silica. Although the expansion of the Ohio brick was greater than that of the Pennsylvania brick, it stood a better load test. The Ohio brick, however, was only burned to cone 14 and if it had been burned to cone 18 the expansion would probably have been less.

The expansion of the silica and magnesite bricks was greater than that of the chrome and fire-clay bricks. There was a large increase in the expansion of the silica brick when the temperature on the outside of the brick was 300° C—probably due to the temporary conversion of cristobalite[2] from the alpha to beta form. The expansion of the magnesite brick was much more gradual with increase in temperature.

[1] *Loc. cit.*

[2] LeChatelier and Bogitch, "The Manufacture of Silica Brick," *Bull. Am. Inst. Mining Eng.*, **141**, 1438 (1918); J. Spotts McDowell, "A Study of Silica Refractories," *Bull. Am. Inst. Min. Eng.*, **119**, 2007 (1916).

FIG 3.—Bricks after testing: (1) Magnesite. (2) Fire-clay. (3) Silica. (4) Chrome.

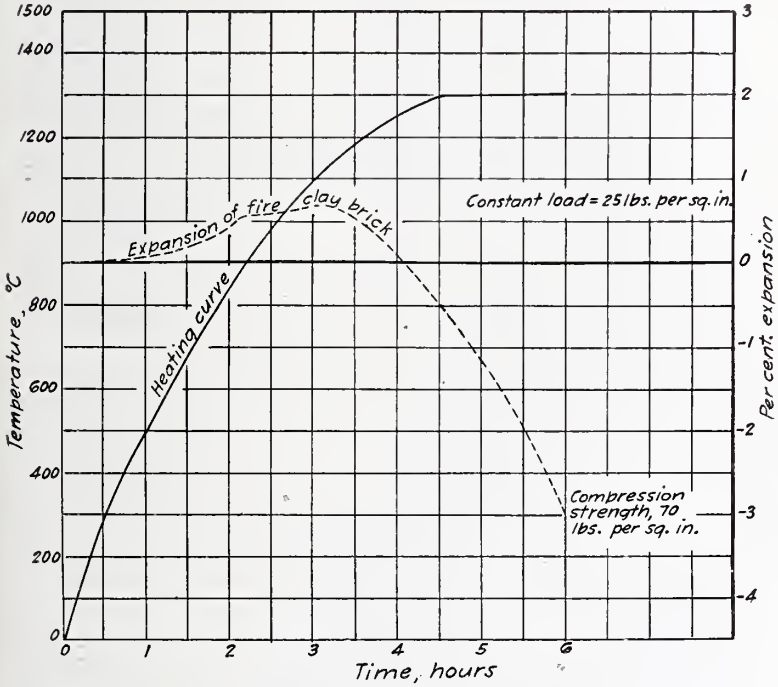

Fig. 4.—Tests on fire-clay brick.

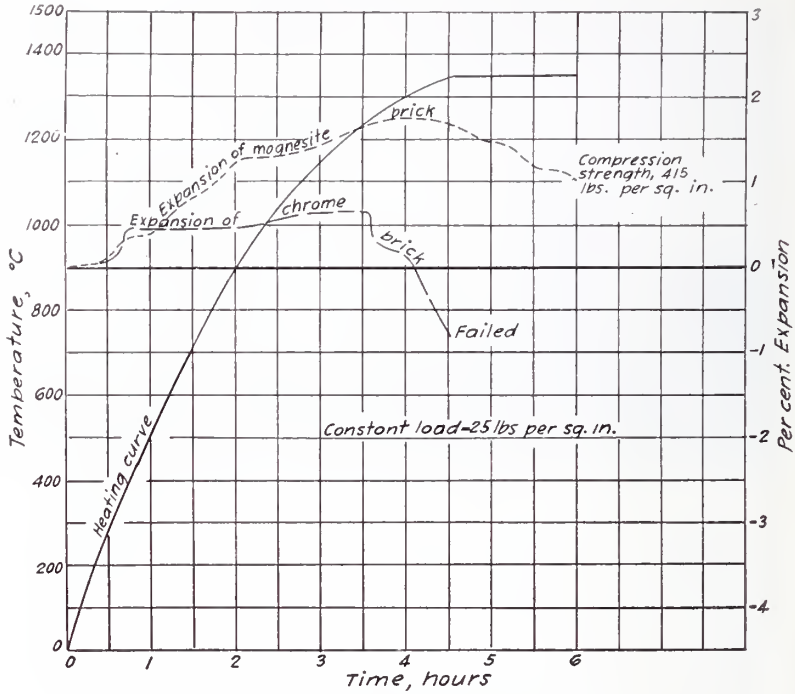

Fig. 5.—Tests on magnesite and chrome bricks.

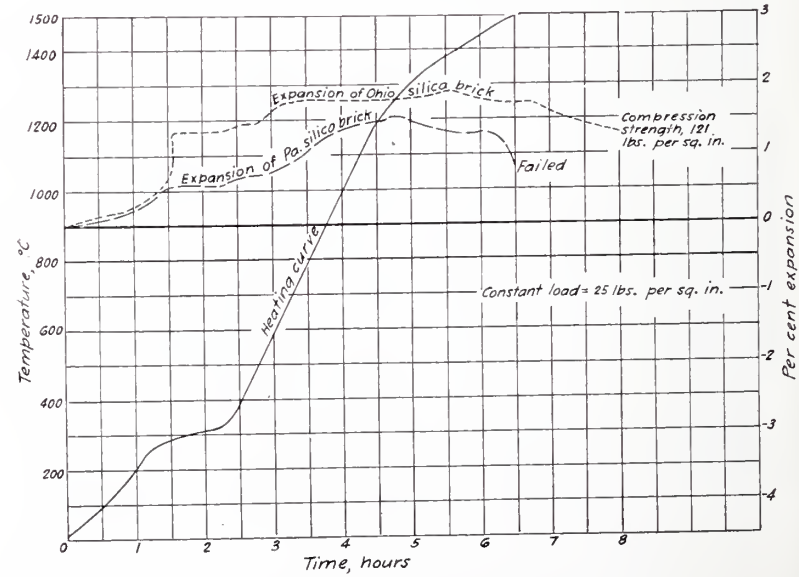

Fig. 6.—Tests on silica bricks.

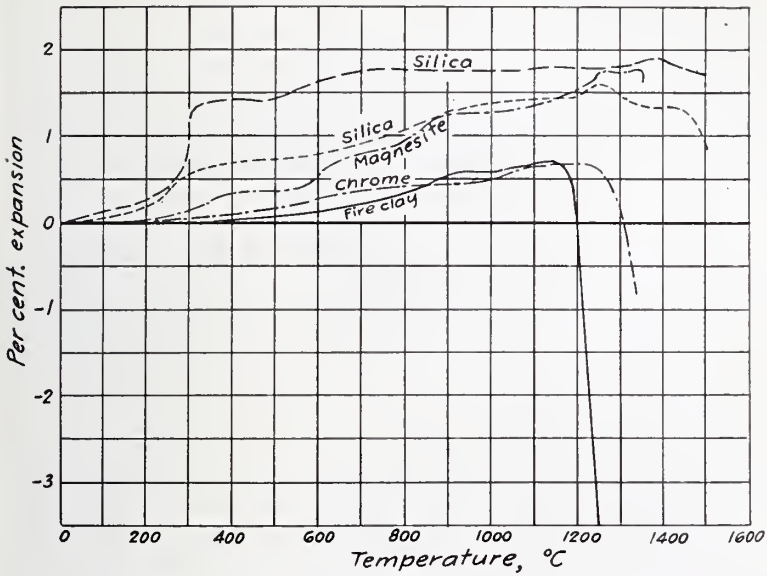

Fig. 7.—Expansion of refractory bricks during load test.

The silica and magnesite bricks show the least deformation while the fire-clay and chrome bricks show the greatest (see Fig. 3).

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THE RAPID ELECTROMETRIC DETERMINATION OF IRON IN SOME OPTICAL GLASSES.

By J. B. Ferguson and J. C. Hostetter.

In the early stages of the development of the optical glass industry in this country, the determination of iron in the materials used, and produced in that industry, was one of the most important chemical analyses made, and was carried out on all raw materials, and frequently also on the glasses. The amounts of iron present in the glasses ranged from 0.01 to 0.02 per cent in the best heavy flints to 0.3 per cent in some experimental glasses, and at times, for purposes of comparison, an accuracy of 0.002 per cent in the determination of this element was desirable. This accuracy can be obtained upon ten-gram samples by the methods based upon the decomposition of the glasses with sulphuric and hydrofluoric acids and subsequent titration of the iron with potassium permanganate, but the use of sulphuric acid solutions necessitates many time-consuming precautions which may not be considered disadvantageous in a complete analysis of a glass with its manifold manipulations, but which should be eliminated if possible when many single determinations for factory control are desired. Fortunately at the time when a need for a more rapid method arose, an investigation of the use of the electrometric method for the determination of the end-points in the stannous chloride-potassium dichromate iron titration was nearing completion at the Geophysical Laboratory and the method was at once applied, first to sands,[1] later to the other raw materials, and finally to the glasses. The information obtained in the last application is presented in this paper.

The description of the electrometric method is in course of publication[2] but for clarity a few details will be repeated here. The principle is simple; the iron, in acid solution, is first reduced

[1] J. B. Ferguson, *J. Ind. Eng. Chem.*, **9**, 941 (1917).

[2] J. C. Hostetter and H. S. Roberts, *J. Am. Chem. Soc.* (Sept. No.)

by stannous chloride and then titrated with potassium dichromate, plotting the electromotive force against the volume of the oxidizer instead of using the inaccurate outside indicator.[1] The curve so obtained undergoes two inflections, the first upon the complete oxidation of the excess stannous chloride, and the second when the iron has similarly become all oxidized; from the location of these inflections the amount of the dichromate used up in the oxidation of the iron may readily be found. A typical curve which indicates the titration of the ferrous iron initially present in the glass in addition to the total iron is shown in Fig. 1. The method provides an accurate means of determining the

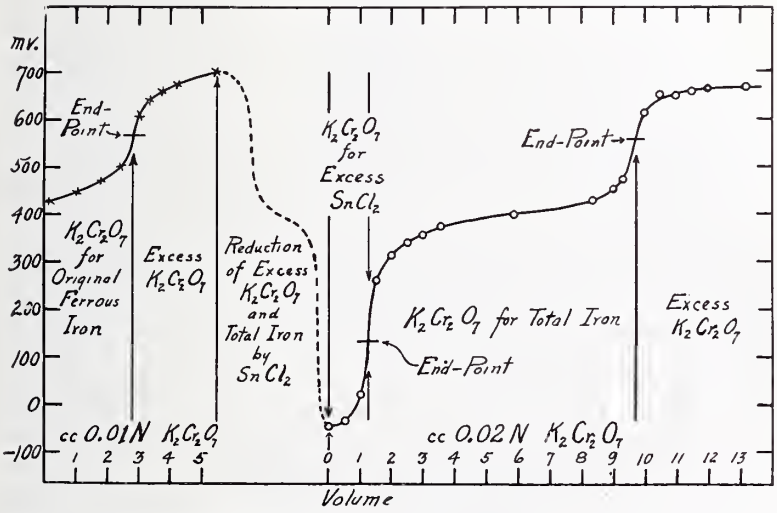

FIG. 1.—Typical curves obtained in the electrometric determination of ferrous and total iron in an experimental glass.

end-points and makes available for accurate work the dichromate titration with its many advantages over the permanganate titration, not the least of which is the use of hydrochloric acid solutions.

[1] Forbes and Bartlett have shown that the use of potassium ferrocyanide as an outside indicator in the dichromate titration may cause an appreciable error. *J. Am. Chem. Soc.*, **35**, 1527 (1913).

The titration is carried out in a flask covered by a "titrating head" which serves the dual purpose of permitting the titrations to be carried out in a current of inert gas and of supporting the calomel half-cell and platinum electrode (see Fig. 2). The elec-

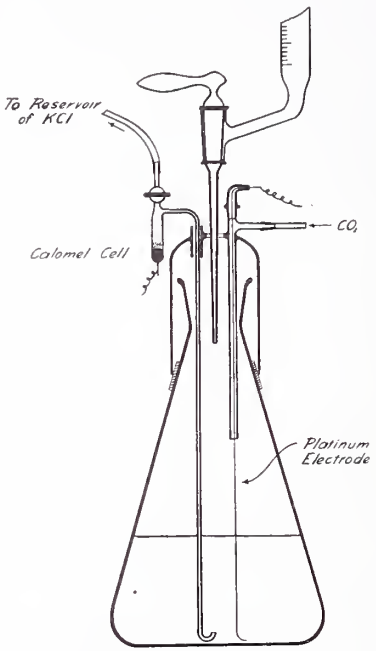

FIG. 2—Flask with "titrating head" in place. The latter carries the electrodes and acts as a cover for the flask during titration.

tromotive force developed is read on a simplified potentiometer designed by Roberts,[1] or by the method used by Hildebrand.[2]

Interfering Substances in the Glasses.—The optical glasses investigated by us were of the following types: Alkali-lead flint, borosilicate crown, light crown and barium crown, and con-

[1] To be described later.

[2] J. Hildebrand, *J. Am. Chem. Soc.*, **35**, 847–871 (1913).

tained only arsenic and boric acid among the substances which might be considered possible interfering substances. Antimony and manganese were not present. Arsenic is, however, reduced to the metal in hot concentrated hydrochloric acid solution by stannous chloride and consequently did not interfere with the determination of total iron. On the other hand, arsenic in the arsenious condition is oxidized by dichromate and therefore interferes with the accurate determination of ferrous iron.[1] Tests carried out on ferric chloride solutions indicate that boric acid also does not interfere; titrations of 6.1 and 6.0 cc. of 0.005 N $K_2Cr_2O_7$ were obtained in two experiments which were identical in all respects but one, namely, the addition of one gram of boric acid to one solution.

Preparation of Samples.—Samples should be pulverized with iron-free utensils such as an agate mortar and pestle. For the preliminary crushing the use of a brass cylinder[2] which loosely fits the pestle materially aids the process and reduces the flying of the glass fragments to a minimum. A powder which will pass 100 mesh is satisfactory.

Hydrofluoric Acid.—Hydrofluoric acid is generally considered responsible for the unsatisfactory end-point often obtained when a permanganate titration is carried out in its presence. To test its effect upon the dichromate titration the following experiments were made. The ferrous iron was determined by titration in solutions containing 100 cc. of 1 : 1 hydrochloric acid, 5 cc. dilute ferrous iron solution, and various amounts of 48-per cent hydrofluoric acid. The solutions were kept in flasks which had by test been shown to yield no appreciable ferrous iron to such solutions under more severe conditions than existed in these experiments and the titrations were made in the cold. The results are given in Table 1.

[1] Smooth curves are obtained when solutions of ferrous iron and arsenious arsenic in 1 : 1 HCl are titrated electrometrically. This is in accord with the well-known ability of certain ferrous salts to reduce arsenic in the higher (arsenic) state of oxidation in hydrochloric acid solution.

[2] Our standard laboratory practice.

TABLE I.

Cc. 48% hydrofluoric acid added.	Cc. 0.01 N $K_2Cr_2O_7$ solution.
0	6.7
5	6.9
10	6.9
15	6.9
25	7.0

These results indicate that in the cold the acid is without effect. In hot solutions such as were used in the analyses of glasses there was frequently a drifting end-point and since its irregular occurrence precluded the placing of the blame for it upon the acid itself, we were led to believe that there must at times be some impurity in the acid capable of being oxidized in hot hydrochloric acid solution by dichromate. The nature of this impurity is somewhat obscure but may well be organic[1] and volatile, because when the acid in question was evaporated alone in platinum no residue remained, but when evaporated with sulphuric acid an appreciable carbonaceous residue was usually obtained. The so-called C.P. hydrofluoric acid usually carries this impurity, so that its presence must be considered in any procedure that is to be of general use.

Glassware Tests.—Good analyses may be carried out in glass vessels in the place of platinum if pure hydrofluoric acid is available. This is possible because the rate of attack of any reagent, as for instance hydrofluoric acid, upon a glass powder,[2] is much more rapid than upon the vessel used as a container, and this is especially true when powdered optical glass is decomposed in flasks of chemically-resistant glass. For this purpose, however, the glass vessel most resistant to hydrofluoric acid would naturally be chosen, and to enable us to make a proper selection the following tests were made:

In each of three Erlenmeyer flasks of identical size (Jena, Fry and Pyrex) were placed 25 cc. of 1 : 1 hydrochloric acid, 5 cc. of

[1] The acid did not behave as though impurities such as sulphur dioxide, arsenious acid, etc., were causing the trouble. See "A Treatise in Quantitative Inorganic Analysis," J. W. Mellor, p. 463, (1913).

[2] Compare the rates of attack found by J. H. Davidson and W. E. S. Turner on glass powders consisting of differently-sized particles. *J. Soc. Glass Tech.* **11**, 284 (1918).

48-per cent hydrofluoric acid and 0.5 cc. of concentrated sulphuric acid and the flasks were set on a hot steam bath for twenty minutes. The solutions were then removed from each flask, separately evaporated in platinum to remove the hydrofluoric acid, taken up in 150 cc. of 1 : 1 hydrochloric, acid and the iron electrometrically titrated after reduction with stannous chloride. The iron (Fe) values obtained were Jena 1.92 mg., Fry 1.85 mg., and Pyrex 1.26 mg., indicating the superiority of Pyrex over the other two types in this respect. These tests were intended as comparative tests and not intended to be in the nature of blank determinations and were purposely carried out under conditions of much greater severity than those existing during an analysis. However, a blank determination for ferrous iron was made subsequently on a Pyrex flask and gave a negligible result.

Analyses in Glassware.—The analyses in glassware were carried out by first decomposing the glass in a Pyrex Erlenmeyer flask and then reducing the iron and titrating in the same flask without the removal of any precipitate which may have formed. In this manner the determination could be carried out in less than ten minutes and the procedure was applicable to those glasses such as flints, borosilicates and crowns which are either completely soluble in a mixture of hydrochloric and hydrofluoric acids or form insoluble precipitates which carry no appreciable quantities of iron. The procedure necessitates the use of pure acids or at least acids upon which definite blank determinations could be made, and usually a slight excess of glass powder was used which insured a minimum attack upon the flask. Comparative results upon an experimental light flint glass analyzed in glassware and also in platinum ware are as follows: In glassware 0.110 and 0.109 per cent iron (Fe), and in platinum ware 0.111 per cent iron (Fe). Most of the C. P. hydrofluoric acid which we obtained was too impure to be used with this procedure for accurate work, but served for the determination of the approximate value of the iron content if the titrations were rapidly carried out since the iron reacted with the dichromate much more rapidly than did the impurity carried by the hydrofluoric acid and thus made possible the locating of the end-point approximately. The curves A and

B, given in Fig. 3, are typical of the effect of this impurity; curve C is a normal curve.

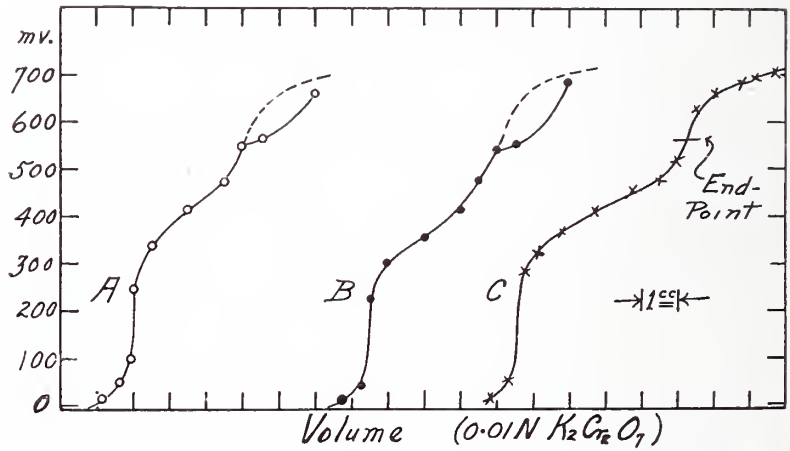

FIG. 3—Titration of total iron. Curves A and B are the result of impurity in hydrofluoric acid used in decomposing the glass. Curve C is a typically normal curve. The end-points for A and B can still be located within a small error.

Platinum Ware.—The platinum used by us was of the purest grade imported before the war. Ordinary platinum as a rule carries an appreciable amount of iron and cannot be expected to yield a constant blank. For this reason only the purest platinum should be used, thus rendering negligible any errors due to the variable amounts of iron obtained from the platinum during different analyses.

Retention of Iron by Precipitates.—The retention of iron by the insoluble salts formed during the decomposition of a glass may be a source of serious error. Allen and Zies[1] state that in some cases as much as 50 per cent of the iron may be so retained. The following analyses confirm their observations. These analyses were made upon a series of light flint glasses which were decomposed with hydrofluoric and sulphuric acids.

[1] *J. Am. Ceram. Soc.*, 1, 739 (1918).

TABLE 2.

Retention of Iron by Lead Sulphate Precipitated in the Decomposition of Light Flint Glasses.

Glass No.	Total Fe.	Fe in lead sulphate.	Per cent total iron in lead precipitate.
43–1	0.105	0.043	41
43–3	0.193	0.117	61
43–4	0.032	0.003	9
43–5	0.099	0.072	73
45–3	0.179	0.049	27
45–6	0.150	0.091	60

The retention of iron (especially ferric) by barium and lead sulphates has been known for some years.[1] Our results given in Table 2 are of interest chiefly because of the variability in the amount of iron so retained. There was some evidence that indicated that this variability was due to the heat treatment of the precipitates inasmuch as more iron was held in those cases in which the solutions were evaporated to dryness than in those in which the evaporation of the sulphuric acid was not carried so far. Lead chloride, on the contrary, was invariably found to be free from even traces of iron, as might have been expected from the crystalline nature of this precipitate.

Total Iron.

The procedures which follow were used by us in some hundreds of glass analyses. The retention of iron by barium sulphate made necessary a separate procedure for glasses containing barium, and procedure (4) was used in place of procedures (2) and (3) when barium glasses were being analyzed. Procedure (1) was not used by us for these barium glasses because of lack of pure hydrofluoric acid. Since barium fluoride is known to be soluble in strong acids,[2] there is no good reason why this procedure should not give good results with these barium glasses.

Procedure (1).—For accurate work pure acids are required. Place 10 g. of glass powder in a Pyrex Erlenmeyer flask and cover

[1] Many references to this subject may be found in J. W. Mellor's, "A Treatise on Quantitative Inorganic Analysis," p. 611 (1913).

[2] Abegg's *Handbuch der Anorganische Chemie* **II**, 2, p. 245.

with 25 cc. 1 : 1 hydrochloric acid. Add not quite enough hydrofluoric acid to decompose all the glass (the amount of hydrofluoric acid required may be calculated approximately and later determined more accurately by test). When the reaction ceases, add enough strong hydrochloric acid to bring the volume to about 150 cc. and the acid concentration to a little above the 1 : 1 ratio and then heat to boiling, reduce with stannous chloride, cool a few seconds and titrate electrometrically with 0.01 N dichromate. Wash several times by decantation with hot water[1] any particles of glass which may have escaped decomposition, transfer to suitable dish, dry over flame, weigh roughly and deduct the weight from the initial weight of charge. The whole operation can be carried out in less than ten minutes.

Procedure (2).—For accurate work with impure hydrofluoric acid. Place 5 g. of glass powder in a platinum dish and moisten with 5 cc. of 1 : 1 sulphuric acid. Add cautiously, a little at a time, 10 to 15 cc. concentrated (48%) hydrofluoric acid. Care must be taken in this operation lest a violent reaction take place and cause a loss of much of the sample. When the reaction has apparently ceased, heat, at first gently and then more strongly, until the sulphuric acid fumes copiously and any organic matter present is decomposed. For this evaporation a Hillebrand radiator[2] may be used to advantage. We used a simpler and cheaper device which worked very satisfactorily. The platinum vessel was placed upon a nichrome triangle which was fitted with silica protecting tubes and the arms of the triangle were bent so that the vessel was held the desired distance above an electric hot plate. Around the triangle and crucible was placed an alundum or similar ring 5–8 cm. in height. After the sulphuric acid has fumed copiously for a few minutes the platinum dish is set aside to cool, and when cold its contents are taken up in 150 cc. of 1 : 1 hydrochloric acid and the iron determined electrometrically in this solution.

Procedure (3).—It sometimes happens when procedure (2) is

[1] This will remove any precipitated lead chloride which may be present.

[2] W. F. Hillebrand, "The Analysis of Silicate and Carbonate Rocks." U. S. Geol. Surv. *Bull.*, **422**, 31 (1910).

used that the organic matter does not coagulate but is suspended in the hydrochloric acid solution in such a fine state of subdivision that it is able to reduce the dichromate and thereby be a source of error. To avoid this, the contents of the platinum dish may be dissolved in just enough hot 1 : 1 hydrochloric acid to prevent the precipitation of the sulphates. This solution is cooled and the insoluble chlorides filtered off and washed several times with cold water. To the filtrate is added a few cc. of 0.1 N alum solution and the alumina and iron are then precipitated hot with ammonia. The iron and alumina are filtered off, washed with hot water and then dissolved with hydrochloric acid from the filter. The hydrochloric acid solution thus obtained is used for the titration. The organic matter either runs through the filter when the iron and alumina are being filtered, or is left on the filter when these are dissolved. The alumina gives more bulk to the precipitate and if but a little is used materially assists in the operation. Suitable blanks must, of course, be made.

Procedure (4).—Since barium sulphate is not appreciably soluble in hydrochloric acid, the iron in this sulphate is lost if procedures (2) or (3) are used with glasses containing barium. The glass may be decomposed as in procedure (2) and the contents taken up in hydrochloric acid as in procedure (3), with the exception that enough acid is used to dissolve and prevent the precipitation of the sulphates other than barium. The solution is filtered hot and the precipitate washed with hot water. The filtrate is cooled and again filtered. This second filtrate contains part of the iron and little, if any, lead or barium. The rest of the iron is in the barium sulphate. The barium sulphate precipitate is ignited in a platinum crucible, fused with sodium carbonate, leached with water, filtered and washed.[1] The sulphuric acid is thus separated from the barium and iron and these are dissolved in hydrochloric acid. The iron from the glass is thus obtained in two acid solutions which can be used for an electrometric titration. They cannot

[1] According to W. F. Hillebrand, when sulphur is determined in rocks by first fusing with sodium carbonate and then leaching with water, a very little iron is to be found in the filtrate with the sulphates. (U. S. Geol. Surv. *Bull.*, 442, 139 (1910). In our experience with procedure (4) the amount of iron in the filtrate was always negligible.

be mixed, else the barium would again be precipitated as sulphate and might remove some of the iron. The titration may be carried out directly, or the iron may be precipitated as in procedure (3), although this would not be necessary in the case of the iron from the barium sulphate, but might be in the case of the other solution.

Ferrous Iron.—Ferrous iron may be determined in glasses provided no other reducing material is present. For this purpose the following procedure was devised, based upon the experiments given earlier in this paper, which showed that hydrofluoric acid and the impurity it usually carries do not react with 0.01 N solutions of dichromate in the cold.

Procedure: 10 g. of the glass powder (glasses other than barium crown) are placed in a Pyrex Erlenmeyer flask with 50 cc. 1 : 1 hydrochloric acid and the acid boiled to expel all air from the glass. The flask is then covered with a funnel through which a glass tube is inserted and by means of which nitrogen or carbon dioxide (freed from oxygen) can be passed through the flask. After the air has been expelled from the flask, 20 cc. of hot concentrated hydrofluoric acid which has previously been boiled for a few minutes to expel the air is added. A violent reaction takes place, and care must be exercised if an accident is to be avoided at this stage. As soon as the reaction is over, enough freshly boiled hydrochloric acid is added to bring the volume to 150 cc. and the acid concentration to a 1 : 1 value. The solution is then cooled in the atmosphere of nitrogen. When cold, the funnel and tube are removed quickly and the titration head inserted in their place. The titrations are then carried out. With the barium crown glasses the procedure is slightly modified because this glass is slightly decomposed by strong hydrochloric acid. The glass powder is first moistened with water (25 cc.) and no acid is added until all the air has been expelled by boiling for some minutes.

Blank Determinations.—The necessity for careful blank determinations is so apparent that it need not be stressed here. A true blank for the total iron determinations would require the use of an iron-free glass or silica powder. This we did not have, and with procedures (2), (3) and (4) we substituted, for the blank,

the iron value obtained when the procedure was carried out without a glass powder. The blank usually obtained in this manner amounted to from 0.2 to 0.5 cc. of 0.01 N dichromate, although in a few cases the hydrofluoric acid carried sufficient iron to materially raise these figures. The blank with procedure (1) was this blank minus the iron value obtained when the sulphuric acid alone was evaporated in platinum and the residue taken up in hydrochloric acid and titrated electrometrically, from which the hydrochloric acid blank had previously been taken. With our pure platinum we found practically the same blank for procedures (1), (2), (3) and (4).

The chemicals and glassware used in the ferrous iron determinations gave no appreciable titration with dichromate, a fact previously mentioned under the discussion of the ferrous iron determination.

The procedures previously outlined were frequently used by us during the years 1917 and 1918 and some of our results have already been published.[1] Our results upon the ferrous iron content of glasses have not previously been published, nor do we find any record of any one having attempted such analyses in the past. It is true that several investigators[2] have studied the relation between the total iron content and the color of certain glasses, and therefore it is the more surprising that no previous attempts have been made to determine the ferrous iron content since the green color so often obtained in many glasses containing much iron is generally ascribed to this ferrous iron. The ferrous iron content of good optical glass is so minute a quantity that any investigation of the relation of ferric and ferrous iron to the transmission of such optical glasses must be carried out upon special glasses containing appreciable quantities of iron. A number of these special iron-bearing optical glasses which had been prepared, were analyzed by us for both ferric and ferrous iron. The relative amounts of ferrous iron were found to vary considerably with the composition of the glass and with several other

[1] Hostetter, Roberts and Ferguson, *J. Am. Ceram. Soc.*, **2**, 356–372 (1919).

[2] C. Dralle, *Chem. Ztg.*, **24**, 1132–6 (1900); R. Zsigmondy, *Ann. d. Phys. u. Chem.*, [4] **4**, 60 (1901).

factors which need not be gone into here but which will be treated in a subsequent paper dealing with these glasses. A few representative analyses will be cited as indicative of the order of magnitude the ferrous iron content may assume in iron-bearing glasses.

TABLE 3.

Total and Ferrous Iron Found in Some Experimental Glasses.

Glass No.	Type of glass.	Total iron as Fe.	Ferrous iron as Fe.	% of total Fe which is in ferrous form.
43–1.....	Light Flint	0.103	0.0112	10.8
43–5.....	Light Flint	0.098	0.0117	12.0
43–6.....	Light Flint	0.101	0.0078	7.7
45–2.....	Light Flint	0.099	0.0207	20.9
45–5.....	Light Flint	0.110	0.0168	15.3
43–2[a].....	Light Flint	0.157	0.0224	14.3
		0.158		
		0.197	0.0241	12.3
41–1.....	Barium Crown[b]	0.138	0.0117	8.5
61–1.....	Barium Crown	0.136	0.0148	10.9
41–2.....	Barium Crown	0.229	0.0334	14.6
61–2.....	Barium Crown	0.230	0.0319	13.8
73–2.....	Barium Crown	0.214	0.0816	38.2
73–4.....	Barium Crown	0.073	0.0280	38.3
49–3.....	Ordinary Crown[b]	0.068	0.0047	6.8
49–4.....	Ordinary Crown	0.078	0.0054	7.0
49–1.....	Ordinary Crown	0.170	0.0109	6.3
85–2.....	Ordinary Crown	0.067	0.0109	16.3
85–5.....	Ordinary Crown	0.125	0.0319	25.5

a The first three determinations (two ferric and one ferrous) upon glass 43–2 by Ferguson and the last two by Hostetter upon different pieces of glass from the same thread-pot.

b The values given for the iron in the ferrous condition in the barium crown and ordinary crown glasses may include some arsenious arsenic due to the presence of this element in these glasses, which were prepared upon the assumption that in glasses all the arsenic exists in the arsenic condition. Allen and Zies have since shown that some arsenic may be present in the arsenious condition. (See *J. Am. Ceram. Soc.*, 1, 739–86 (1918).)

Summary.

The results of the application of the electrometric determination of iron with stannous chloride and potassium dichromate are discussed in this paper. The electrometric method enables one to

make rapid and accurate analyses for both ferric and ferrous iron provided interfering substances are absent. Under favorable conditions such an analysis can be made in ten minutes and may be carried out in glassware. Four different procedures are described for total iron and one for ferrous iron. A number of analytical results, including many ferrous iron determinations, are given. The ferrous iron content of the glasses proved to be dependent upon a number of factors and in some cases reached values in excess of 35 per cent of the total iron present.

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July, 1919.

A STUDY OF HIGH-FIRE PORCELAINS.

By Chi C. Lin.

The rapid growth of the American chemical-porcelain industry, as a result of the World War, has given an impetus to the American clay manufacturers generally and is of good omen—particularly to the domestic production of all kinds of high-fire porcelains. That this problem holds the important place in the field of ceramic researches it really deserves is evident from the recent augmentation of technical literature on the subject. This investigation was undertaken with the hope of contributing some useful data regarding certain porcelain bodies fired to a comparatively high temperature.

A study of true porcelains, as manufactured in China, Japan and continental Europe, brings out the fact that the greatest manufacturing losses are due to sagging or distortion of the ware in firing. The practice of maturing glaze and body together makes the proper supporting of the ware during the vitrification stage very difficult, as the glaze adheres to any support and the points of contact appear as faults in the finished ware. Excessive warping is prevented to some extent by placing each article in a separate setter, but the cost of production is thus materially increased and the difficulty only overcome in part.

In the manufacture of most American china, the desired vitrification is secured in the bisque burn, during which the ware is embedded in sand, ground kaolin, or flint. Thus supported, the ware attains the necessary degree of vitrification without excessive warping. The glaze is then applied and matured at a temperature (generally not exceeding cone 5) sufficiently below the vitrification temperature of the body so that no danger of warping is encountered in the second or glost burn. However, the degree of translucency noted in the true porcelains of Europe and Asia is not obtained in the softer-fired wares of the United States. Moreover, the glazes used on the soft-fired porcelains are less

desirable on account of their inferior hardness. They also possess smooth, glossy surfaces which are not so attractive as the egg-shell surfaces of the high-fire true porcelain glazes.

The question of producing a high-fire porcelain by a modification of the American method, that is, by raising the glost temperature to permit the use of a hard-porcelain glaze, seems to offer promise, because, thereby, the danger of warping can be reduced to a minimum, while the desired translucency and glaze texture can also be secured. To accomplish this, the body must be of such composition that it will mature at relatively the true porcelain temperature—at least cone 13. This is the minimum temperature at which crystalline sillimanite forms in quantity in porcelains and, we are told, produces the fine blue-white translucency of the true porcelain. If the glaze be applied and matured at the necessary temperature it will then bring out its beauty. This glaze must be of the true unfritted porcelain type, free from lead oxide, the maturing temperature being safely below that at which the body will begin to distort. Its composition must also be such as to warrant a satisfactory glaze-fit.

The first problem is, therefore, to produce a body having excellent working qualities in the plastic state and one which will mature with the minimum amount of warping at a temperature sufficiently high to secure the desired translucency. The second problem is to fit the body with a glaze having the qualities of a true porcelain glaze, but which will mature at a temperature such that it can be fired below the deformation point of the body.

Previous Research on this Subject.

An investigation as to the effect of composition on the strength of porcelain bodies has been reported by Lester Ogden.[1] A. S. Watts has reported some expansion data secured on European porcelains.[2] R. C. Purdy and A. P. Potts have reported on the coefficients of expansion of white-ware mixtures.[3] E. T. Montgomery and M. G. Babcock have reported an investigation on chemical porcelain[4] and state that this product, which deforms

[1] *Trans. Am. Ceram. Soc.*, 13, 395 (1911).
[2] *Ibid.*, 13, 406 (1911).
[3] *Ibid.*, 13, 431 (1911).
[4] *Ibid.*, 18, 88 (1916).

at cone 25, must be fired sufficiently high (at about cone 16) to become vitreous and translucent, and that the glaze used must be a hard porcelain one maturing at about the same temperature.

The European low-bisque and high-glost-fire bodies are high in clay, vitrification being secured by high temperature of firing and not by fluxes. Under the microscope these bodies are found to be free from undissolved silica and bonded into a strong mass by sillimanite needles. F. H. Riddle and W. W. McDanel, in reporting an investigation of some types of porcelain,[1] compare European and American pottery practices and the properties of typical and special porcelain bodies. A. V. Bleininger[2] observed that the temperature treatment plays an important rôle in the development of porcelain and that firing temperatures higher than cone 10 are conducive to a more homogeneous structure.

The constitution and microstructure of porcelains have been studied by Zoellner,[3] Plenske,[4] Klein,[5] and Peck.[6] Klein found that in both high-quartz low-feldspar and low-quartz high-feldspar bodies, burned at 1310° C (cone 9), the original clay dissociates into both amorphous and crystalline sillimanite, the former greatly predominating. Quartz is only slightly soluble. At higher temperatures (1380°–1400°) amorphous sillimanite disappears and gives rise to the crystalline form. Quartz, also, dissolves to a considerable extent. Klein concluded that the constitution and microstructure of porcelains depend upon the temperatures of burning and vary with the temperature changes.

The time of burning is a factor of as great importance as that of the burning temperature in determining the constitution and microstructure of the ware. Microscopic examination of porcelains by Peck brought out the effect of time and temperature on the microstructure. Bodies fired for longer periods at lower temperatures were produced which simulated very closely those fired at higher temperatures for shorter periods.

[1] *J. Am. Ceram. Soc.*, **1**, 606 (1918).
[2] *Ibid.*, **1**, 697 (1918).
[3] Sprechsaal, *Yahrgang*, **41**, 471 (1908).
[4] *Ibid.*, **20, 21, 22** (1908).
[5] *Trans. Am. Ceram. Soc.*, **18**, 377 (1916).
[6] *J. Am. Ceram. Soc.*, **2**, 175 (1918).

It must be borne in mind, however, that in commercial practice the fuel consumed in maintaining a low temperature for a long period of time would exceed that consumed in raising the temperature sufficiently high to develop the microstructure of the porcelain in a shorter period. Hence, from an economic standpoint, high-temperature treatment should be resorted to for the production of true porcelains.

Investigation.

The present investigation covers twenty-nine porcelain bodies having the following limits of composition:

Feldspar	10–20 per cent
Flint	30–45 per cent
Clay	30–45 per cent
Ball clay	10 per cent

The range of composition of the mixtures is shown in the triaxial diagram (Fig. 1). It will be noted that the field includes

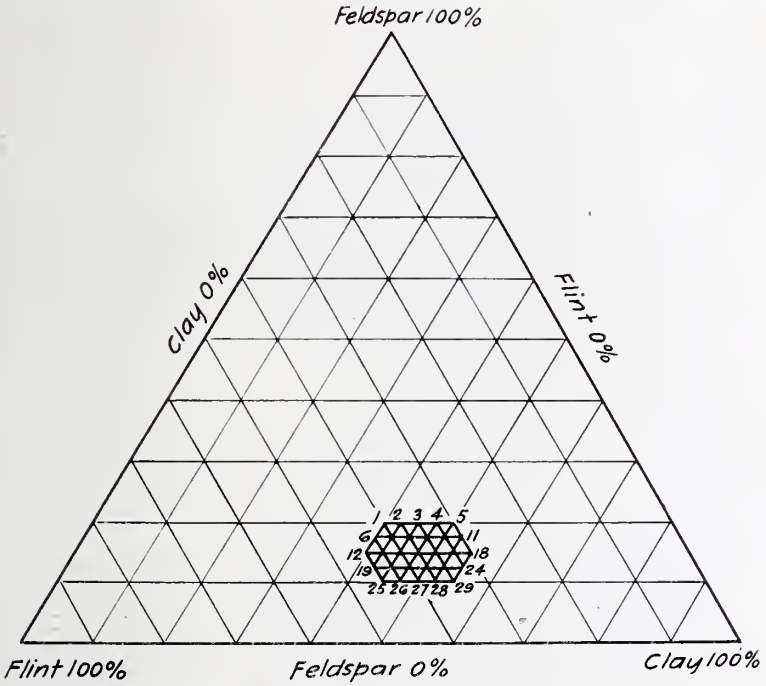

FIG. 1.—Field of body compositions covered.

ordinary white-ware and chemical-porcelain bodies. The chief purpose of this study was to determine the common physical properties of these bodies when fired to cone 10 and cone 13–14.

Raw Materials.—The non-plastic materials used in the bodies were Canadian feldspar and Ohio Silica Co. flint. The clay used consisted of a mixture of 10 per cent Mayfield ball clay and equal portions of McIntyre, Georgia kaolin and Edgar, Florida kaolin. The chemical compositions of these materials are given in Table 1.

TABLE 1.

	Canadian feldspar.	Ohio Silica Co. flint.	McIntyre, Georgia kaolin.	Edgar, Florida kaolin.	Mayfield, Kentucky ball clay.
SiO_2	64.00	99.18	44.90	45.67	61.23
Al_2O_3	19.61	trace	38.49	38.45	25.05
Fe_2O_3	0.43	0.26	0.76	0.75	1.30
TiO_2	...	trace	1.20	0.10	1.35
CaO	0.76	trace	...	...	0.10
MgO	...	trace	...	0.05	0.20
Na_2O	2.46	...	0.30	0.06	1.90
K_2O	12.78	...			
Ignition	...	0.11	...	...	8.35
Total H_2O	...	...	14.35	14.86	...
Combined H_2O	...	...	13.55	13.36	...

Preparation.—Five kilograms of each body mixture was ground in a ball mill for five hours with sufficient water to produce a slip suitable for casting. This slip was passed through a 150-mesh sieve and allowed to stiffen on a plaster slab to a plastic mass suitable for molding and jiggering.

Test Pieces.—Trials in the form of bars, cups, wedges and cubes were made. For each body mixture, eight bars 7″ long and having a 1″ square cross-section (all measurements of trials were made in the plastic state), were made in a brass mold. A shrinkage mark 10 cm. long was scratched on each bar. Ten cups, approximately $2\frac{1}{2}$″ in diameter by $2\frac{1}{2}$″ high, were made by casting and ten, approximately $3\frac{1}{2}$″ in diameter by 2″ high, were formed on the jigger. The wedges, made by pressing in a plaster mold, were 3″ long, 1″ wide, and tapered from $\frac{3}{4}$″ to 0″.

The cubes measured 1″ on each side—the corners having been well rounded off by hand.

Drying and Burning.—The trials were first dried at room temperature and finally in a gas-dryer. The cups were finished with a sponge.

Three trials of each shape were fired to cone 10 in a down-draft laboratory kiln. Of these, one cup made of each body mixture was glazed with a porcelain glaze maturing at the same temperature as the body so that a color comparison of the finished ware could be obtained. The other trials were unglazed. A

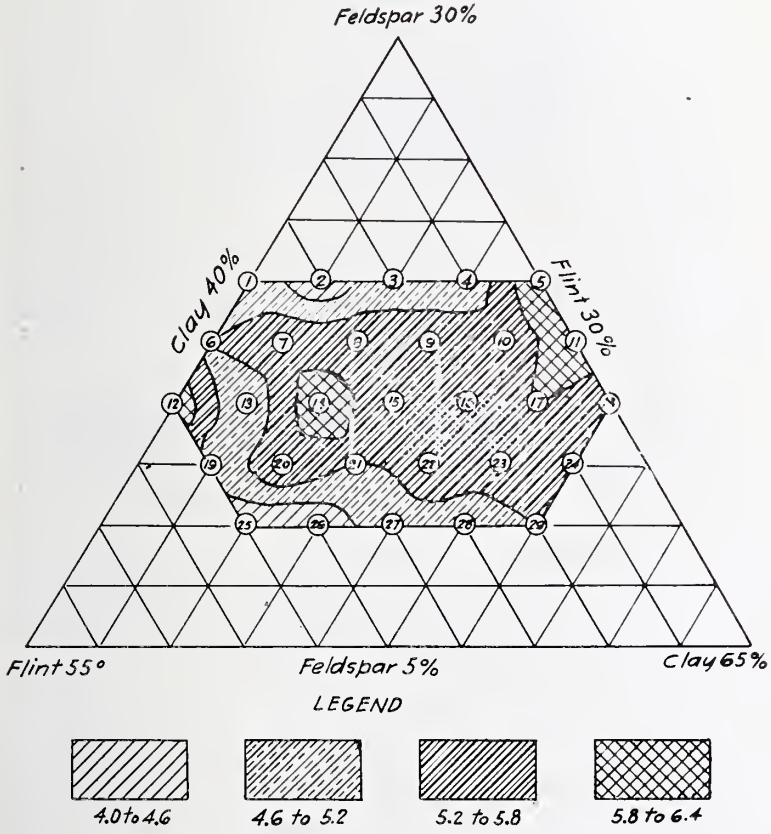

FIG. 2.—Drying shrinkage in per cent of wet length.

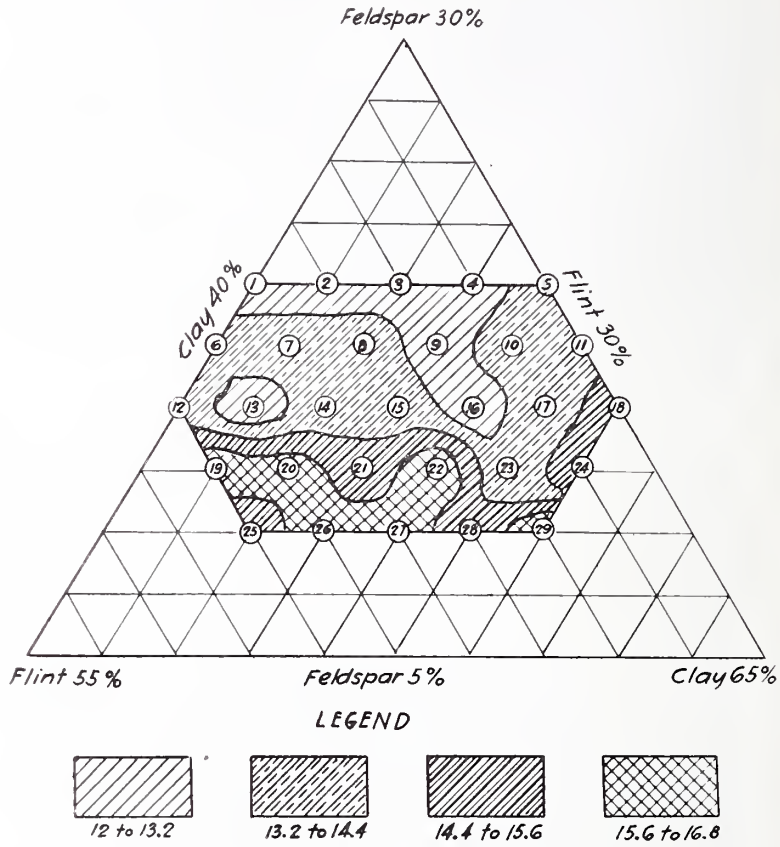

FIG. 3.—Total shrinkage in per cent of wet length.

similar group of trials was fired at cone 13–14 in the same kiln. Cone 10 was reached in twenty-eight hours and cone 13–14 in thirty-two hours, the temperature gradient near the completion of the burn being approximately 20° C per hour.

Shrinkage Measurements.—The drying and burning shrinkages of the bars were measured and expressed in per cent of the lengths of the wet pieces (see Figs. 2 and 3 and Table 2). The burned bars were broken in a transverse-strength machine having knife edges five inches apart. Both the average and maximum moduli of rupture are given in Table 3 for comparison. The maximum

TABLE 2.

Body No.	Percentage composition.			Per cent drying shrinkage.	Per cent total shrinkage.		Per cent absorption	
	Feldspar.	Clay.	Flint.		Cone 10.	Cone 13–14.	Cone 10.	Cone 13–14.
1....	20.0	40.0	40.0	4.8	13.1	12.7	3.45	0.0
2....	20.0	42.5	37.5	4.6	12.4	12.3	2.72	0.0
3....	20.0	45.0	35.0	5.1	14.6	12.7	2.36	0.0
4....	20.0	47.5	32.5	5.0	13.2	12.5	3.08	0.0
5....	20.0	50.0	30.0	5.9	15.4	14.0	3.64	0.0
6....	17.5	40.0	42.5	5.2	12.7	14.3	4.64	0.0
7....	17.5	42.5	40.0	5.3	13.2	14.0	5.03	0.0
8....	17.5	45.0	37.5	5.4	13.0	13.3	5.34	0.0
9....	17.5	47.5	35.0	5.5	14.5	12.5	5.34	0.0
10....	17.5	50.0	32.5	5.4	13.6	14.0	5.20	0.0
11....	17.5	52.5	30.0	6.3	13.8	14.0	4.09	0.0
12....	15.0	40.0	45.0	6.2	13.6	13.7	4.44	0.0
13....	15.0	42.5	42.5	4.9	12.1	12.3	4.63	0.0
14....	15.0	45.0	40.0	6.2	13.7	14.3	7.52	0.0
15....	15.0	47.5	37.5	5.7	13.0	13.5	5.45	0.0
16....	15.0	50.0	35.0	5.4	14.3	13.0	6.82*	0.0
17....	15.0	52.5	32.5	5.8	15.0	14.3	1.40*	0.0
18....	15.0	55.0	30.0	5.5	15.7	15.3	4.19*	0.0
19....	12.5	42.5	45.0	5.0	14.3	15.8	4.93*	0.40
20....	12.5	45.0	42.5	5.25	14.0	16.0	4.78*	0.57
21....	12.5	47.5	40.0	5.2	13.2	15.5	3.61*	0.0
22....	12.5	50.0	37.5	5.4	13.2	15.7	5.61	0.04
23....	12.5	52.5	35.0	5.3	12.5	14.0	5.27	0.08
24....	12.5	55.0	32.5	5.5	13.2	15.5	5.24*	0.02
25....	10.0	45.0	45.0	4.2	11.8	15.2	9.90*	1.08
26....	10.0	47.5	42.5	4.25	12.5	16.2	9.35*	1.58
27....	10.0	50.0	40.0	5.0	13.2	16.0	9.40*	1.02
28....	10.0	52.5	37.5	4.8	11.5	15.0	7.85	0.82
29....	10.0	55.0	35.0	5.2	13.0	16.5	6.90	0.71

* These figures were interpolated from the actual data obtained as given in Table 4.

moduli of rupture are plotted in Fig. 4. The broken bars were weighed dry and then soaked in water over night. After boiling for one hour, the wet weight of each sample was obtained. From the dry and wet weights of the same sample, percentage absorption

TABLE 3.

Body No.	Modulus of rupture in lbs. per sq. in.				Warpage in inches of deflection. Cone 13–14. 5″ span.	Translucency in mm.		Rattler loss in per cent. Cone 13–14.
	Cone 9–10.		Cone 13–14.			Cone 10.	Cone 13–14.	
	Aver.	Max.	Aver.	Max.				
1....	4737	5900	3347	4200	0.10	2.3	2.8	3.7
2....	6203	6500	6227	6850	0.05	2.5	3.0	5.5
3....	4840	5940	6100	6800	0.0	2.3	2.8	5.0
4....	6820	7350	6957	7460	0.0	2.5	2.8	8.0
5....	4703	5370	7053	8350	0.0	2.5	2.8	9.4
6....	5893	6250	8660	9390	0.09	2.0	2.8	1.40
7....	5743	6030	7940	8210	0.03	2.3	2.7	2.8
8....	4913	5320	8970	10000	0.03	2.0	2.8	3.1
9....	5237	6040	5900	7620	0.05	1.8	2.8	6.0
10....	6373	7350	8917	10220	0.02	2.0	2.5	4.5
11....	5283	5940	7640	8330	0.10	2.0	2.5	4.0
12....	5697	6400	6723	8330	0.10	2.0	2.5	1.7
13....	5607	5800	8233	8420	0.06	2.3	2.8	2.7
14....	4930	5800	9410	10470	0.06	1.8	2.5	2.8
15....	5147	5580	8350	9050	0.08	1.5	2.0	1.6
16....	3700*	4320*	8753	9250	0.07	1.8	2.3	3.6
17....	5200*	5810*	9697	11550	0.05	1.8	2.3	0.0
18....	2500*	2550*	7980	9350	0.08	1.8	2.0	0.2
19....	3030*	3450*	7743	10160	0.0	1.8	2.0	0.0
20....	4300*	4540*	9703	10450	0.06	2.0	2.2	0.4
21....	5590*	5950*	9127	11400	0.0	2.0	2.3	0.4
22....	3890	4300	8670	11280	0.0	1.8	2.0	0.25
23....	2925	3160	11045	11140	0.0	1.8	1.8	0.7
24....	3320*	3860*	9567	10920	0.0	1.7	1.8	1.0
25....	2360*	3200*	6767	7330	0.0	1.4	2.0	0.4
26....	2400*	3100*	7960	8440	0.03	1.5	2.2	0.1
27....	3080*	3760*	7987	9720	0.0	1.4	1.6	0.25
28....	2553	2900	8783	9620	0.0	1.3	1.6	0.4
29....	2960	3600	9707	11200	0.0	1.3	1.6	0.1

* These figures were interpolated from the actual data obtained as given in Table 4.

was calculated on the basis of the dry and wet condition (see Table 2 and Fig. 5). In the cone 13—14-burn, two bars of the same size as above were supported on two clay wedges, having an

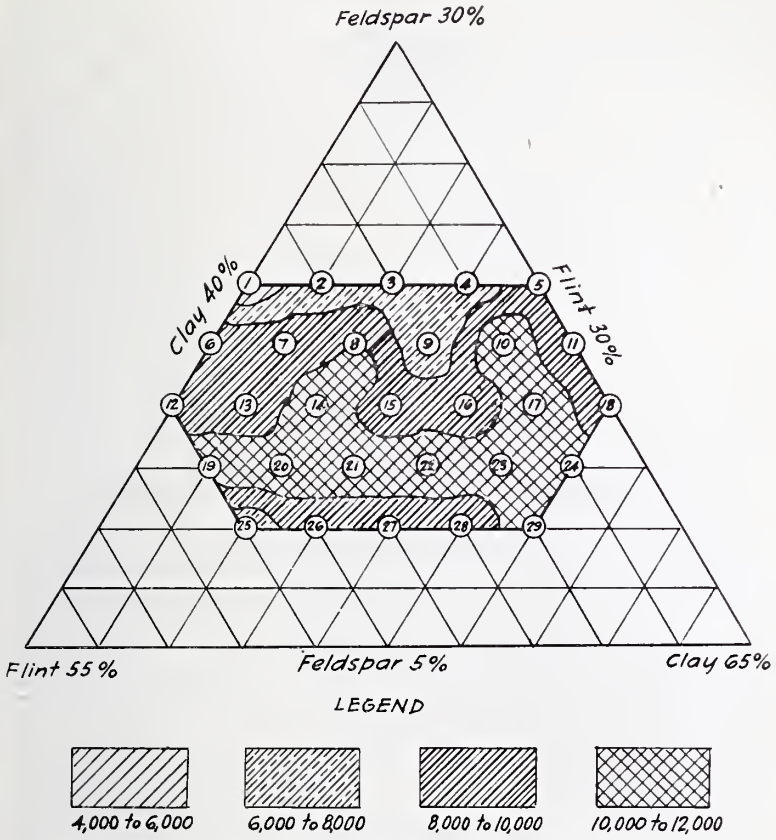

FIG. 4.—Maximum moduli of rupture (cone 13–14) in pounds per square inch.

equilateral triangular cross-section, and placed five inches apart in the sagger. The deflection in inches was gauged as a measure of warpage (see Fig. 6).

Translucency.—For the determination of translucency, the wedges made of different body mixtures were arranged in a row (in good light). A metal wire, about 1 mm. in diameter, was held close to the lighted side of the wedge under examination. The thickness of the wedge, at the point below which the wire became invisible, was measured by means of an Ames dial.

TABLE 4.

Body No.	Firing temperature.	Per cent absorption.	Modulus of rupture.	
			Average.	Maximum.
16.....	Cone 11–12	1.66	3913	4450
17.....	Cone 11–12	0.38	5095	5780
18.....	Cone 11–12	1.14	2185	2820
19.....	Cone 11–12	2.08	3117	3550
20.....	Cone 9	5.79	3750	3960
21.....	Cone 9	4.38	4870	5180
24.....	Cone 9	6.35	3205	3970
25.....	Cone 11	8.02	2460	3360
26.....	Cone 11	7.71	2493	3210
27.....	Cone 11	7.76	3187	3880

The measurements are given in Table 3 and plotted in Fig. 7.

Resistance to Abrasion.—Since an apparatus for the standard rattler test, as specified by the American Society for Testing Materials, was not available, a modified method from which comparative data of the resistance to impact were obtained, was employed. Two cubes of each body composition were weighed and placed in a ten-gallon ball mill (total weight = 1345 gm.). Forty flint pebbles, from $1^1/_2''$ to $2''$ in diameter, weighing 4265 gms., were added and the ball mill run for one hour at thirty-five revolutions per minute. The cubes were then sorted out, dusted, and weighed separately. The loss in weight divided by the original weight gave the percentage rattler loss. The results obtained are tabulated in Table 3 and plotted in Fig. 8.

The study has proven so extensive that a discussion of the glaze will be left to a later date.

Conclusions.

From the data given in the above tables and plotted in the accompanying diagrams, the following conclusions may be drawn:

1. The drying shrinkage and total shrinkage of the bodies studied show great irregularity. Very broadly speaking, however, they increase with a corresponding increase in clay content.

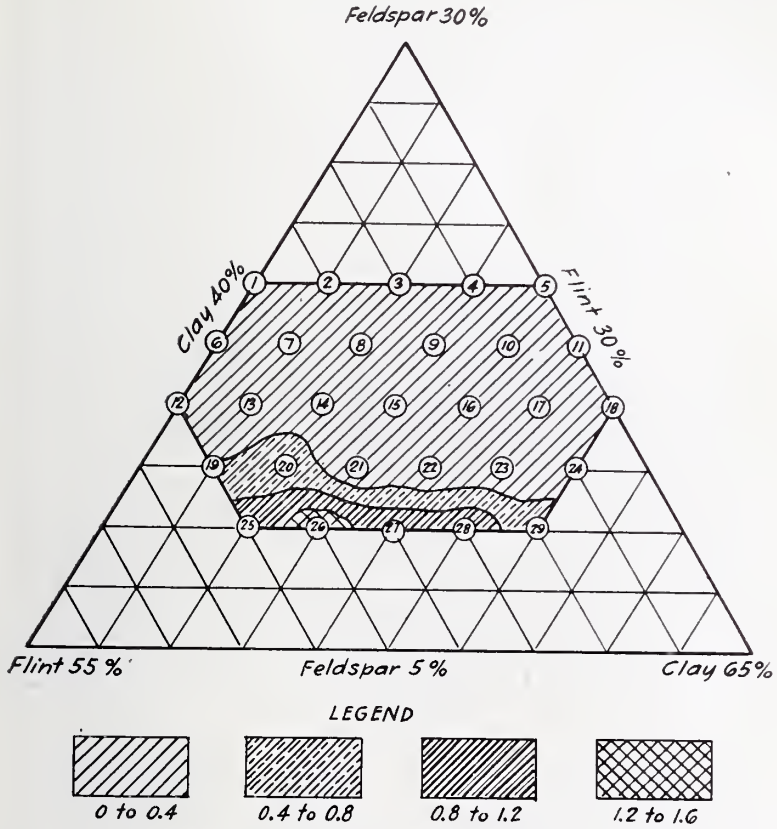

FIG. 5.—Absorption (cone 13–14) in per cent of burned weight.

2. With bodies 1, 2, 3, 4 and 5, which contained 20 per cent feldspar, the total shrinkage was smaller at cone 13–14 than at cone 10. This was due to the development of a vesicular structure in the bodies fired at cone 13–14, as was apparent in the cross-section of the broken bars. The little material increase of cross-breaking strength gained by raising the firing temperature from cone 10 to cone 13–14 points to the fact that these bodies were overburned. Bodies 9, 16, 17 and 18, likewise, did not shrink as much at cone 13–14 as at cone 10. With the small number of trials made, the author is not able to explain the phenomena at present.

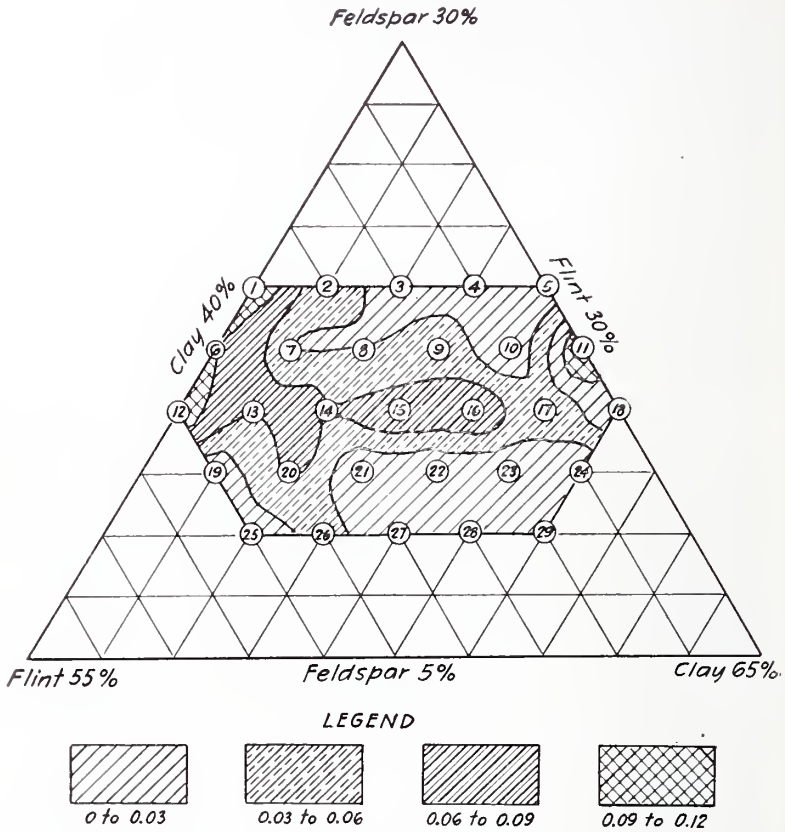

FIG. 6.—Warpage (cone 13–14) in inches of deflection (5″ span).

3. At cone 10 none of the bodies were thoroughly vitrified—as indicated by the considerable amount of water absorbed. All of the bodies containing from 15 to 20 per cent feldspar became non-absorbent at cone 13–14. The bodies containing 12.5 per cent feldspar absorbed a small amount of water, while those containing 10 per cent feldspar absorbed considerably more.

4. A considerable increase in cross-breaking strength was noted in the bodies, other than the first five mentioned above, when fired at cone 13–14. Generally speaking, there was a greater increase in strength in bodies low in feldspar and high in clay, the feldspar being more active.

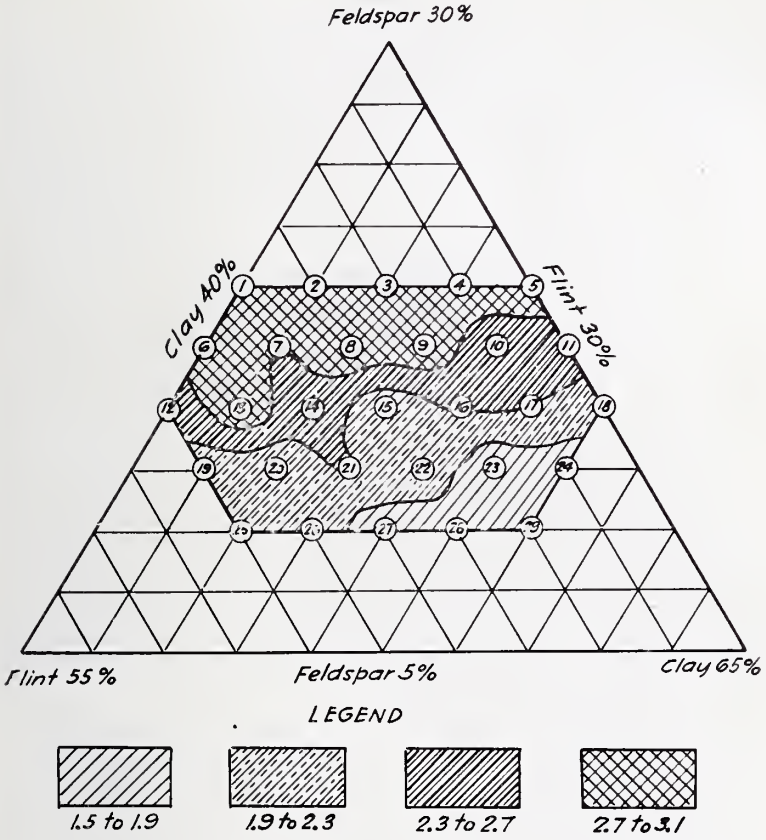

FIG. 7.—Translucency (cone 13–14) in mm. of light penetration.

5. The warpage of the bodies at cone 13–14 showed very irregular variations and, as a whole, was not excessive.

6. The bodies fired at cone 10 were semi-translucent, while those fired at cone 13–14 showed distinct translucency, so far as both depth of light penetration and quality are concerned.

7. The color of the bodies was decidedly improved by firing at a higher temperature. The cone 13–14 product was a beautiful bluish-white, while the cone-10 product appeared creamy. In the bodies fired at cone 13–14, there was a noticeable tendency to a pinkish tinge as the content of feldspar was decreased.

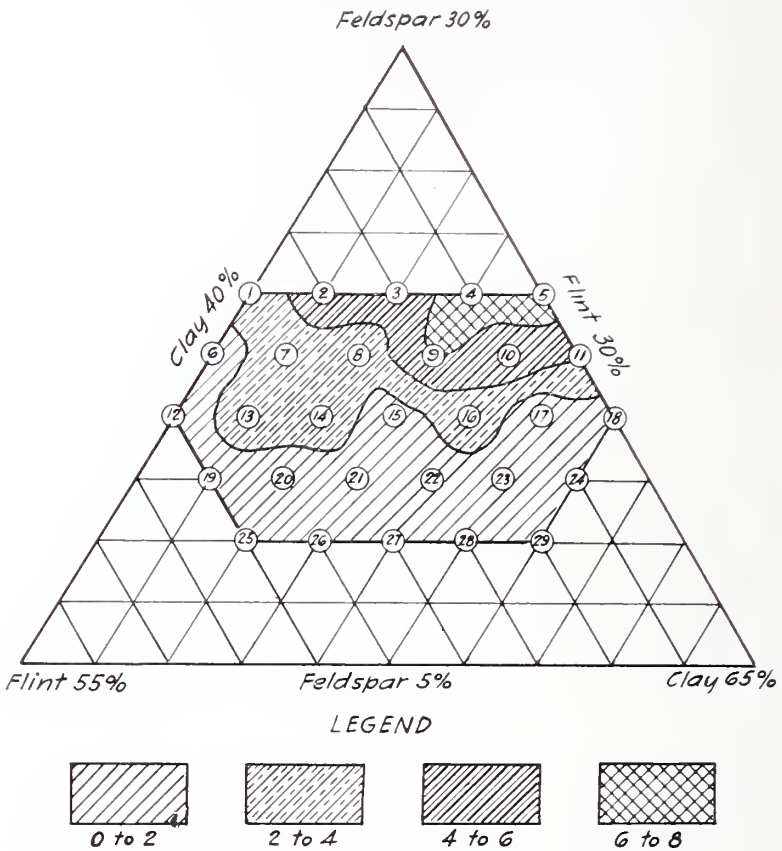

FIG. 8.—Rattler loss (cone 13–14) in per cent of original burned weight.

8. The results of the rattler tests were consistent with the observations of Lester Ogden as given in his paper.[1] Toughness is apparently dependent more on the amount of flint present than on either feldspar or clay. A comparison with the data on moduli of rupture shows that the bodies having high cross-breaking strength, as would be expected, suffered no or little loss in the rattler test.

9. Taking all the above properties into consideration, bodies 8, 17 and 21 were the best of those fired at cone 13–14.

[1] *Trans. Am. Ceram. Soc.*, **13**, 395 (1911).

Acknowledgment.

Prof. Arthur S. Watts has given valuable advice during the progress of the investigation. His assistance and encouragement made the work an instructive as well as enjoyable undertaking. To him the author wishes to express his appreciation and acknowledge his obligation.

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THE MANUFACTURE OF ABRASIVE WHEELS.[1]

By S. C. Linbarger.

When civilization reached the stage of advancement where man began to form weapons and the implements necessary for his existence from hard metals instead of stone, he came to realize the distinct advantages which could be gained if it were possible to smooth the surface and sharpen to a keen edge the weapon so crudely fashioned. It was only the natural sequence of events which led to the discovery of a substance supplied abundantly by nature, which when rubbed over the surface of the metal would abrade it and gradually wear it away. Although the abrasive properties which some of these rock formations evinced were greatly inferior to others, they gained widespread use owing to their geologic occurrence and to the suitability of other of their physical properties. The ultimate result was that natural sandstone attained a paramount position in the abrasive field which it held for many years.

Even the introduction and perfecting of the artificial wheels in which grains of natural minerals were used did not materially affect the status of the natural grindstone. However, the latter is now being rapidly superseded, even in the small village blacksmith shops, by grinding wheels made of artificial abrasive material.

This of course only follows in line with a similar replacement which took place in the large metal-working factories several years ago. They were the first to realize the decided advantage to be gained in amount of production and accuracy of finish obtained by using grinding wheels which could be made with physical characteristics applicable to their individual grinding conditions.

The advent of better and more accurately constructed machines having interchangeable parts and the desire to produce them as rapidly and economically as possible, has been an influential factor in the development and the universal adoption of the artificial

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abrasive wheel as a necessary adjunct to present day metal-working equipment. Especially is this true in the automotive industry which demands standardized parts in enormous quantities. The quantity-production of Liberty-motor parts which was reaching such large proportions during the latter days of the World War, to say nothing of truck and tractor production throughout the entire war period, would have been impossible without the artificial grinding wheel and the modern grinding machine. There is not one of the multitude of parts in an automobile but whose production is directly or indirectly influenced by the efficiency of electrically-formed abrasives. And it can be safely stated that without them the price of automobiles today would be prohibitive to all except the very rich, and the number of farm tractors in use could be stated in hundreds instead of thousands.

Nearly a half-century ago the desire for a better grinding medium led the pioneers in the industry to seek some satisfactory means of bonding the hard, sharp particles of emery and corundum into an integral grinding wheel. Owing to the hardness of the grains of the material and the ability to control the coarseness and density of the finished product within certain limits, it rapidly replaced the natural stone in places where its initial cost was not a deciding factor.

When carborundum was discovered several years later, its value as an abrasive was immediately apparent owing to the extreme hardness and sharpness of the solid plate-like crystals unlike any found in nature. However, it was several years before it could be manufactured cheaply enough to warrant its use other than in the polishing of rare gems. When electric furnaces were devised and designed wherein this material could be made in sufficient quantities and at such cost of production that it could be profitably used as an ingredient in commercial-sized grinding wheels, the question of binding the crystals into an integral wheel arose. The adoption of fusible clays as a matrix for securely bonding the crystals was immediately recognized and practical wheel-makers, who had gained experience in the bonding of natural emery, set about to produce wheels composed of bonded silicon-carbide crystals. Their efforts were crowned by the production

of a satisfactory wheel which proved greatly superior to natural abrasives in many grinding operations. At first the production was necessarily limited to this one class of wheel. As its introduction into the machine shops of the country became more universal, the need for wheels which would cut faster and cooler and give greater production on certain classes of work presented complex problems which involved laborious and extended ceramic research.

The ingenuity of the men to whom this work was entrusted soon asserted itself and it was but a short time until the ceramic problems involved in bonding carborundum had reached a high state of perfection. At the outset it was seen that carborundum was not adapted to all classes of grinding, as even the natural abrasives proved more efficient grinding mediums on the hard, high-tensile-strength materials. But owing to the non-uniformity in physical properties and chemical composition of the natural abrasives, great difficulties were encountered in bonding the grains and producing wheels of uniform quality from time to time. This led to the development of an artificial aluminous abrasive which was much superior in cutting properties to natural emery and could be made uniformly from day to day. Combinations of bonding materials which were adaptable to carborundum were not suitable to this new aluminous abrasive. Neither were those which had been used successfully with corundum entirely satisfactory, consequently new processes of manufacture and bonding had to be worked out. As soon as the high efficiency of grinding over turning was demonstrated and the modern grinding machine was introduced, the new and varied uses to which grinding wheels were applied necessitated a scientific study of the desirable qualities which a grinding wheel should possess for certain factory conditions and various classes of work. This research has continued unabated until at the present time the grinding-wheel manufacturers are in a position to furnish wheels of the proper grit and grade and the proper density and toughness of bond to meet the individual grinding conditions of the consumer.

When it is realized that the speed of the wheel, the speed of the work, the type of machine, and the physical properties of the material to be ground affect the rapidity of cut and the life of the

wheel, it is apparent that it is not possible at the present time to produce a universal wheel for grinding a given class of material. For instance, manganese steel as produced by different companies for different purposes might vary as much as ten per cent in the manganese content and have such vastly different physical properties that the type of wheel which would be suitable for the high-manganese steel would be wholly unsuited to grinding the other. Again, one plant might desire to grind the fins and risers from large electric-steel castings. As these sharp promontories exert considerable dressing action, the grinding wheel must be tougher and harder than one used on steel of the same composition but where the castings are light in weight. In the snagging operations, heat generated in the grinding operation is of little or no consequence, while in the case of finish grinding, cutting without the generation of heat is the chief desideratum. Therefore, wheels of different density and bond structure must be supplied to successfully meet the conditions encountered.

These are only a few of the innumerable instances that could be cited wherein the abrasive-wheel manufacturer is compelled to produce a product that will meet the demands of the customer and he must further be able to duplicate the wheels which are supplied on succeeding shipments. This duplication resolves itself into maintaining a uniform strength of bonding material, the same hardness or grade of wheel, and simultaneously keeping a uniform density of wheel structure.

The bonding of abrasive wheels can be grouped under three general classes, depending upon the bonding material used, namely: vitrified, silicate and elastic. In the case of vitrified bonds, the binder consists of a clay or clays which fuse at commercial kiln temperatures. In the silicate type of wheel, the bonding matrix is principally sodium silicate or commercial water-glass combined with chemical substances which produce an insoluble silicate at low temperatures. The organic binders may be any insoluble organic compounds but the principal ones now used are shellac and rubber.

Artificial abrasives are manufactured at temperatures only obtainable in electric furnaces. In the case of carborundum the crystalline material is formed by passing an electric current

through a mixture of sand, sawdust and coke which is packed in a long, rectangular resistance-furnace. By the utilization of an enormous amount of electrical energy, the temperature of the furnace is brought up to the temperature at which the silica is volatilized. In this gaseous state one molecule of carbon combines with an equivalent amount of silicon, forming upon cooling a core of pure crystallized carbide of silicon between the electrodes, which are located in opposite ends of the furnace. As this material is a definite chemical compound, the crystals always have the same physical properties.

Aluminous abrasives are formed by the conversion of an amorphous aluminous substance into a crystalline aggregate of alumina crystals cemented together by a matrix of refractory oxides. This change is brought about by the fusion of the crude material, which occurs in nature as the mineral bauxite, in an electric furnace of the arc type. By the exercise of accurate chemical and electrical control during the furnacing process, it is possible to produce aluminous abrasive-grains of various degrees of hardness and toughness which make them peculiarly applicable to certain classes of grinding operations.

After cooling, the furnaces and pigs are carefully stripped of all unconverted or unfused mix and the suitable material is crushed to a size convenient for transportation to the grinding-wheel factory. The lumps are then crushed in powerful jaw crushers and further reduced in successive rolls which gives a range of sizes which are suitable for use in abrasive wheels, sharpening stones, refractories, etc. Dust, dirt and other foreign material, which may have been introduced in the handling and crushing of the crude material, is removed by thoroughly washing the grains by hydraulic means. It then passes through a dryer which removes all traces of moisture. Particles of iron and other magnetic contaminations are removed by passing the material over a magnetic machine. The grains are then accurately graded by passing them over screens of the proper mesh. The range of grits used is from those which will pass an 8-mesh screen to those of almost impalpable powder. The fine powders which cannot be accurately graded by screens or cloths are separated by hydraulic classifiers

which are similar in construction to the elutriators as used in determining the fineness of grain of clays.

Vitrified wheels are made by two general processes known respectively as the pressed and puddled process. In the pressed process the procedure is to weigh out a given amount of the desired abrasive grains of the proper gradings and to this is added a certain percentage of the bonding ingredients. The amount and character of the various clays used depend, of course, on the desired physical properties which it is wished to impart to the finished wheel. The components are then thoroughly mixed in a dry condition in a mechanical mixing device after which enough water is added to make the mass adhere. The consistency obtained is about like that used in a dry-pressed brick. The material is evenly distributed in a mold which is of slightly larger dimensions than the size of the desired finished wheel or stone. The mold is then placed in a hydraulic press and subjected to a high pressure. The amount of pressure applied depends upon what characteristics and grades are to be developed in the ultimate product. The green wheels next go into a drying chamber or dry house where they are dried very slowly. They remain here until they are thoroughly dry and in a proper condition to be handled and set in the kilns.

Upon delivery to the kiln room, each wheel or abrasive stone is carefully inspected for molding- or drying-cracks and only those which show no defects are allowed to go into the kilns.

In the puddled process, the accurately weighed grain and bond are placed in large mixing kettles and a sufficient quantity of water is added to bring the mix to a thin slip condition. When the ingredients are thoroughly mixed, the slip is drawn off into molds which are somewhat larger than the desired wheels. The puddled wheels, as they are designated, are placed in dry houses until they become sufficiently dry to handle. They are then removed from the molds and go to machines where they are cut to within a fraction of an inch of the proper diameter, thickness, and arbor hole; the additional stock being allowed for final finishing and trueing after coming from the kilns.

From this point on the two processes converge and the method

of completing the finished products is the same as that employed in the manufacture of pressed wheels.

The kilns used are the ordinary type of pottery kiln and the ware is placed in saggers and set in stacks or bungs in a manner similar to that employed in the burning of the more delicate ceramic wares. The kilns are fired to a temperature sufficiently high to thoroughly vitrify or flow the bonding materials. Coal is the fuel used and, owing to the high temperatures required to mature the bond and to the large mass of material which must be uniformly heated to this temperature, the length of time required for a complete cycle of the kiln is about two weeks.

As the name implies, the bond ingredient of vulcanized wheels is rubber. The process employed in the manufacture of this type of wheel is similar to that which is used in the fabrication of all rubber products. The proper proportions of abrasive grits and high-grade crude rubber are weighed out and enough sulphur to insure proper vulcanization is added. The ingredients are then mixed by repeatedly passing the mass through steam-heated rolls. After a homogeneous mixture is obtained, the material is passed through finishing rolls which are spaced so that the material issues from the opposite side in a sheet which is the thickness of the desired wheel. The slabs are then cut into discs of the correct diameters and having the proper sized arbor holes. The green wheels are then given a careful heat treatment to insure proper vulcanization of the bond.

Silicate and shellac-bonded wheels are formed by intimately mixing the respective ingredients with grain and forming in molds by the application of pressure. They are baked in ovens at low temperatures, the degree of heat being just sufficient to impart the requisite strength or bonding properties to the bond.

The wheels go from the kilns and ovens to the finishing department, where the crust or scale developed in the burning process is removed from the sides or face of the wheel and it is brought down to the proper thickness and shape desired. This is accomplished by passing a hardened steel dresser or a diamond across the flat face of the wheel while it revolves in a lathe-chuck.

The wheels are again inspected very rigidly for uniformity of mix, proper fusion of bond, and defects due to burning and hand-

ling. They are also carefully tested at this time for proper grade. *Grade* is a term used by abrasive manufacturers to designate the relative hardness of an abrasive product. It is usually designated by a letter or number, the most common method being to use the letters of the alphabet. Some companies use the first letters to distinguish the harder grades while others use just the reverse. Grade is often erroneously looked upon as an absolute value. Although every precaution is taken by the manufacturer to maintain a strict technical control over every ingredient and step in the process which enter into the manufacture of his product, it is impossible to do more than establish certain limits within which the hardness of a wheel must fall to be of a given grade.

The grade of a wheel is determined by the resistance which it offers to the penetration of a small chisel-shaped tool which oscillates about its longitudinal axis through an arc of about 90 degrees. In order to maintain a uniform product and to keep within the prescribed limits, reference and comparison are frequently made with a set of standard grade-pieces which are kept for that express purpose. If a wheel proves to be of a harder or softer grade than the markings on it would indicate for which it was made, it is rejected by the inspector and scrapped. If it is satisfactory in all respects, it proceeds to the bushing department where a lead-, or in some cases, steel- or babbitt-bushing is put in the wheel, with a hole or arbor left in it which is a few thousandths of an inch larger in diameter than the spindle on which the wheel is to be operated.

The next operation is to bring the wheel down to the proper diameter. This is done by revolving it on a spindle and passing a diamond or wheel-dresser across the peripheral face. The finished wheel proceeds to the balancing ways where it is tested for absolute balance. A wheel out of balance would cause endless trouble in most grinding operations, with a consequence that wheels must be in perfect balance when they leave the factory.

In order to determine conclusively that the bond has sufficient tensile strength to withstand the centrifugal forces set up when the wheel is in operation and that there are no invisible defects in the structure, the wheels are next given a speed test. This consists in rotating the wheels on a spindle which is connected with

a main driving-shaft by means of cone pulleys so that any desired speed can be obtained. The wheel is encased in a steel lined compartment so that in case breakage should occur, the pieces are confined. The recommended speed for vitrified wheels is from 5000 to 6000 surface feet per minute. In order to insure complete safety in operation the wheels are speeded to approximately fifty per cent above the recommended operating speed. The order number and test number, with the speed at which the wheel has been tested, are recorded and the workman who speeded the wheel is required to go before a Notary and swear to the truthfulness of the speeds which he has recorded.

The wheels after leaving the speeder are again carefully inspected for defects in workmanship and go to the expert graders who pass on the hardness or grade. All satisfactory material goes to the shipping room where it is packed carefully in sawdust, to prevent breakage in transit, and is then ready to go to the ultimate consumer.

When it is realized that abrasive wheels must be made in all sizes from the very minute points which are used by dentists to wheels 36″ in diameter and 12″ thick, and meet all the conditions encountered in grinding practically everything from feathers to the hardest alloy steels, and give desired finishes on work which will embrace everything from snagging castings to putting an edge on razor blades, one can readily understand the innumerable classes of materials which the grinding-wheel manufacturer is compelled to produce and duplicate from time to time.

Carborundum Co.,
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THE EQUIPMENT OF A CASTING PLANT FOR THE MANUFACTURE OF GLASS POTS.[1]

By Frank H. Riddle.

During the past two years the Clay Products Section of the Bureau of Standards has developed methods for the manufacture of glass pots by the casting process. The object of the present paper is to describe the methods used and proposed plans for a plant suitable for casting ten 600-pound glass pots per day.

The general plan for such a plant is shown in Fig. 1. The different units are so arranged that the materials and products move continuously forward through the factory and there is no interference between the various processes and units.

Storage and Preparation of Pot Materials.

The first unit is that used for the storage and preparation of pot materials and is shown in the upper left portion of Fig. 1. Along the outer wall of the room, facing the railroad siding, are the storage bins for raw, unground materials. The capacities of these bins will depend upon the composition of the pot body. About half the total capacity will be used for grogs and the remainder for ball clays, kaolins, feldspar, etc.

The equipment for this unit consists of a bench A, for cleaning pot shell, jaw crusher B, dry pan C, elevator screen and return chute D, magnetic separator M, elevator E and dump car F running on the track G over the ground-material storage bins. The raw materials are transferred from the storage bins by wheelbarrow or conveyor to the crusher or to the dry pan. All material that passes through the dry pan is passed over the screen D. The properly-sized materials may be passed over the magnetic separator M or sent directly to the elevator E which unloads into the dump car. The tailings are returned directly to the pan to be more finely ground. If the grog is to be sized it is necessary,

[1] By permission of the Director, Bureau of Standards.

of course, to use several screens in a set; separating the various sizes after they pass through the screens. Experience has shown that the grog should be accurately sized and also be passed over the magnetic separator. Magnetic separators for dry-ground materials are now on the market and although the first cost is high the better quality of the product warrants the investment.

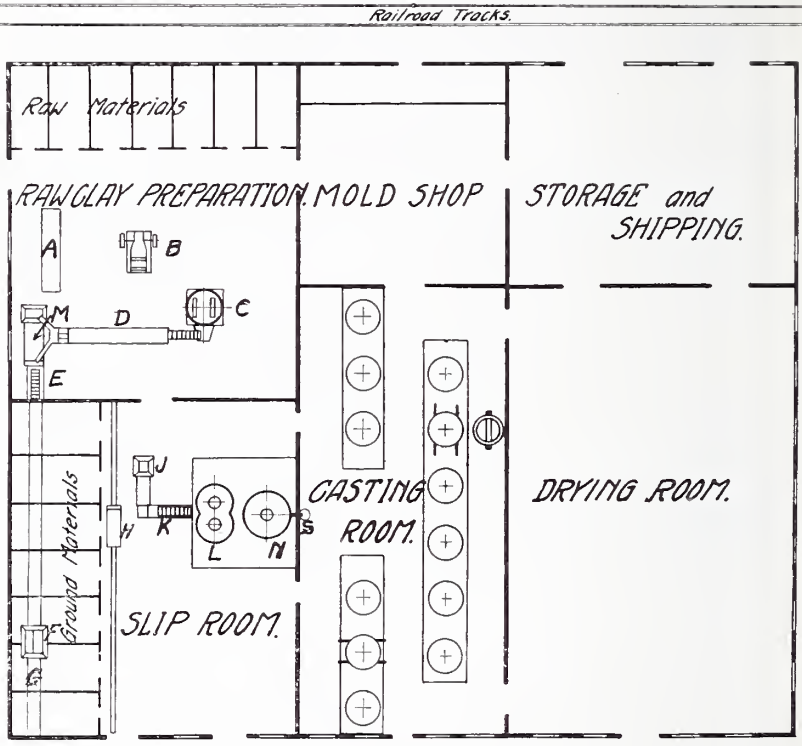

FIG. 1.

Preparation of the Casting Slip.

The next unit of the factory is the slip-preparation room. The ground materials are shoveled from the bins into a scale car H (Fig. 1) which runs on a track as indicated. The size of this car will depend upon the size of the blunger. Regardless of the size of the car, all of the materials should be added in the proper

proportions to make the batch weight used. The materials are transferred from the scale car to a dry mixer J, from which they are discharged into the boot of the blunger-charging elevator K. A continuous mixer should be used. The mixed materials are raised into the blunger L. It is desirable that the blunger be located at a height sufficient to allow the slip to flow by gravity into an agitator, N, then over a 4-mesh screen four feet above the floor of the casting room.

The blunger may be of either the single- or double-paddle type, but the latter is preferable. Both vertical shafts should revolve in the same direction to give the best results. If a single blunger type is used, the lining should be shaped so as to form a hexagonal interior rather than cylindrical, as this tends to prevent the mass of thick slip from rotating as a unit. In a double blunger the lining should extend from the center of each side into the space not swept by the shaft arms—thus preventing the slip from accumulating at these points. The surface of the lining should be made as smooth as possible to facilitate cleaning.

The body should be blunged at least three hours. Somewhat longer than this is preferable; in fact, it is better to use a blunger large enough to contain sufficient slip for an entire day's run. A blunger with a slip capacity of 680 gallons will hold sufficient slip for casting ten 570-pound pots. It should have a slightly larger capacity, however, to allow for loss.

If the blunger is of sufficient capacity the agitator may be dispensed with. The blunger, and agitator if used, should be covered to keep the slip clean and to prevent excessive evaporation of water. Large ports and open troughs should be used for emptying out and transporting slip so that they can be cleaned easily. The discharge pipe or trough from the slip room into the casting room should be placed at a height such as to permit easy operation by a man standing on the casting-room floor. Time is thus saved in filling the carrier bucket. The feed troughs and agitator should be thoroughly washed whenever necessary and a settling tank be provided to catch any grog in the wash water so as to prevent it from clogging the factory drain system.

A body of the following composition has proven satisfactory for optical-glass pots, both from the view-point of the casting be-

havior of the slip, and that of burning the pots and melting the glass:

Material.	Per cent.
Whiteware bisque grog	48
Ball clays	18
Kaolins	28
Feldspar	6
	100

The specific gravity of the slip is maintained at about 1.90. The salts added amount to 0.2 per cent of the total weight of the dry materials. For this particular body, equal amounts of sodium silicate and sodium carbonate are used.

The method of charging the blunger is as follows: The required amount of water is first introduced and the sodium silicate added. The body materials are then introduced, although only about one-half the total batch is charged at first, and allowed to blunge for a few moments until well mixed. The amount of each successive addition of body material is less than the previous addition. The length of time required for each charge to become thoroughly mixed with slip will, of course, increase as more material is added and the mixture thickens. The entire time of charging is about one hour. When this is completed, the sodium carbonate solution is added and the blunging continued. The consistency of the slip should be carefully determined from time to time. This may be judged by feel and by the weight per pint, or by means of a flow-viscosimeter. The latter should have a capacity of at least one gallon and an efflux not less than $^3/_4$ inch in diameter.

The Mold Shop.

Before going to the casting unit the mold shop will be described at this point. As shown in Fig. 1, the mold shop is located at the end of the casting room so that the casting-room bridge-crane can be used both in making and distributing the molds. The mold shop should be at least as wide as the crane-way and not less than fifteen or twenty feet long. A longer room will permit of the storing of blocks, cases, spare molds, plaster, etc.

The mold can be made by actual turning on a horizontal wheel, similar to the surface grinder used for grinding sanitary ware. The diameter of the wheel must be as great as the diameter of the pot mold. Provision should be made for mixing sufficient plaster to make an entire mold-section at one pouring. The grinding wheel may be omitted if the molds are built on a flat-top table with sweeps, as is the practice in the making of terra-cotta molds.

The shape of the mold is very important and the design should be carefully worked out. The size of the belt, slope at the under edge, and other details should be accurately determined.

The mold consists of four parts, as shown in Fig. 2; namely, base, shell or case, ring, and core. The shell must be tapered to allow lowering away from the pot and base which are supported on the pedestal. The core must taper sufficiently to permit lifting up from the cast pot through the ring. The shell and ring should both be well bound with steel bands. The mold rests on a wooden frame which is long enough to span the floor trench and wide enough to allow clearance between the two side members of the frame for it to fit down over the pedestal. However, the clearance should not be greater than necessary, since the base piece of the mold must rest on the frame and the two cross pieces which hold the frame together. The core is clamped down into the shell and ring by means of a cross plank which is bolted on both ends to the support frame (see Fig. 2). A funnel with three openings leading down through the ring into the mold should be provided. The leads should be spaced at equal distances and be two or more inches in diameter. All metallic parts through which the slip flows should be made of galvanized iron.

When the experimental work in the casting of glass pots was first started it was thought necessary, for the continuous operation of a casting plant, to have duplicate sets of molds so that one set could be used one day and the duplicate set the next day while the first set was drying. A year's experience, however, has definitely proven that it is possible to use the same set of molds continuously through the week, allowing them to dry out over Sunday. This saving in molds, space and equipment is of considerable importance from an operating standpoint. To facilitate this daily operation it is advisable to have the walls of the molds

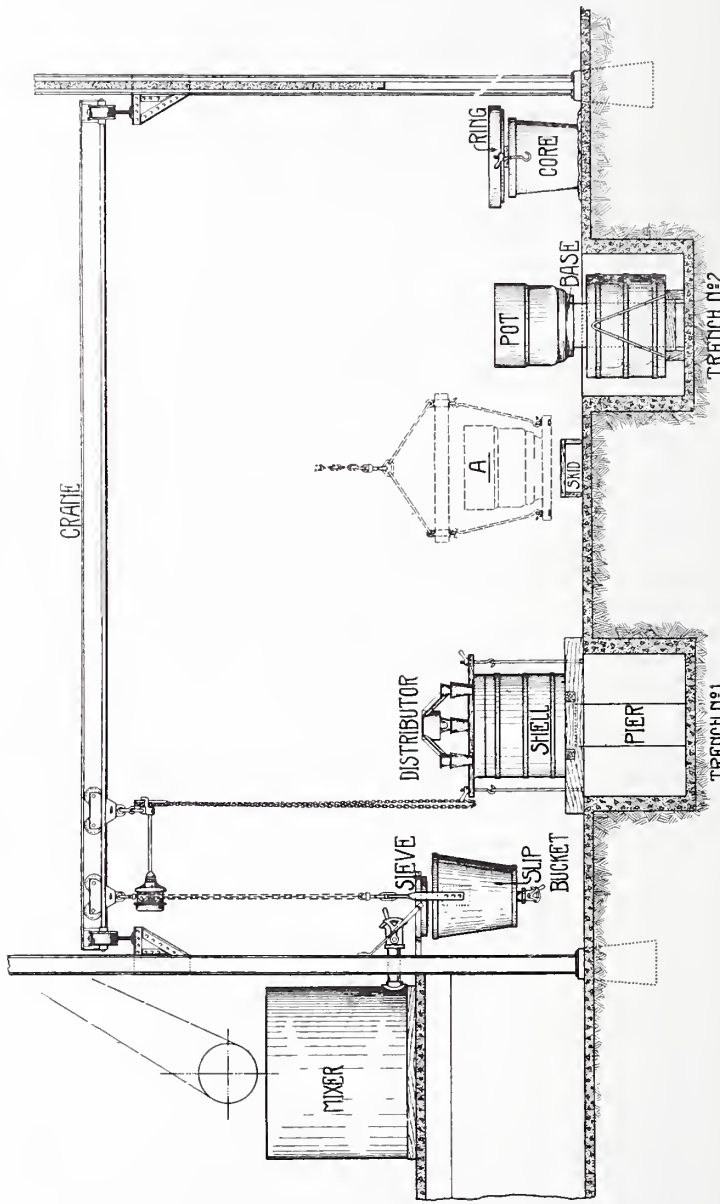
CRANE
RING
CORE
POT
BASE
TRENCH N°2
A
SKID
DISTRIBUTOR
SHELL
PIER
TRENCH N°1
SIEVE
SLIP
BUCKET
MIXER

FIG. 2.

as thin as possible without detriment to their strength. Also, to maintain the temperature of the casting room at from 60° to 70° F during the day time and from 70° to 80° F at night, at the same time keeping the air well circulated. If these conditions are maintained the water absorbed from the slip by the mold is evaporated from the surface of the mold as rapidly as it collects and the work can go on continuously. If the room is cold and damp and the walls of the molds too thick, it will be necessary, after the molds have become saturated, to stop work until they can again be dried out. These precautions also apply to the casting of any kinds of ware in plaster-of-paris molds. The strength of a thin mold can, of course, be very much increased by the use of bands or other outside supports. Reinforcements may also be used in the mold but the former methods are preferable.

Casting.

The casting room is arranged (see Figs. 1 and 2) with a traveling bridge-crane covering the entire area of the room. The crane should not be heavier than necessary and a one-ton chain block is sufficiently strong. The ease of working could be facilitated by having the room six or eight feet wider, by placing a row of posts down the center and by using two cranes, each covering half the width of the room. This would necessitate a trough for carrying the slip to the far crane but this could be accomplished. The crane is arranged with slings; one for picking up the entire mold; one for picking up the pot; and one for carrying the slip bucket.

The floor space is arranged so that the molds are set up in two longitudinal rows. There is sufficient space between the side walls and the line of molds to permit the placing of the cores and rings on the floor when removed from the molds after casting and enough additional space to permit handling the case. An aisle is left between the two lines of molds wide enough to allow space for handling the pots and trucking them away. Space should be provided at each end of the room to permit passage of the pots into the dryer room at the side. There should be a break in the center of the line of molds, on the side near the agi-

tators, so as to allow the slip-carrying bucket to be run in under the slip spouts (See Fig. 1).

As shown in Fig. 2, there is a depressed floor or trench under each line of molds. This is deep enough to permit lowering the case mold and its carriage so that the top is below floor level. A series of piers or pedestals is built along the center line of each depression and spaced longitudinally so that their centers coincide with the centers of the casting molds when in position. These pedestals are of a height such that they project above the floor level about 4″ less than the height of the mold-support timbers.

The operation of filling the molds is simple. The slip bucket, supplied with an outlet beneath, is placed under the agitator-tank outlet and filled with slip (it is best to pass the slip through a coarse screen). The filled bucket is lifted and carried by the crane to a proper filling position over the mold. The filling should be continuous to prevent the trapping of air. The air outlets or issue holes, cut in the core where it fits into the ring, should be large enough to permit the slip to flow as rapidly as possible from the tank. The entire operation of filling should not require more than 10 minutes.

The filling should all be done through one port and the slip not allowed to flow into the other ports until the mold is full. Several casts were made with the slip flowing down the three ports at the same time and joining at the center in the bottom. In this case the areas of contact of the slip coming from each port failed to join thoroughly and defects resulted. Fig. 3 shows two sides of one of these weak unions—sufficiently weak to permit the glass to eat entirely through (note the flow lines of slip in the body).

When sufficient slip is poured in to run well up toward the top of the funnel, there will be enough to complete the casting, allowing for the settle of the slip due to absorption. The height of the funnel above the mold and the resulting pressure affect the casting. This height of supply and the pressure produced are very important points and the life of a pot is largely determined by the pressure used.

The length of time of casting is governed by the kind of slip and the dryness of the mold. It will probably vary from 16 to 24 hours. With a 16- to 18-hour cast, the system works very well

as the molds can be filled the latter part of the afternoon and emptied the next morning.

The mold has an air-inlet plug in the center of the bottom of the core. This can be reached from the top and the hardness of the cast can be determined by removing the plug and feeling the clay body. The thickest part of the pot and the last to cast solid is at this point.

To remove the pot from the mold, the funnel and leads are first taken off. By unscrewing the two lever nuts and unhooking

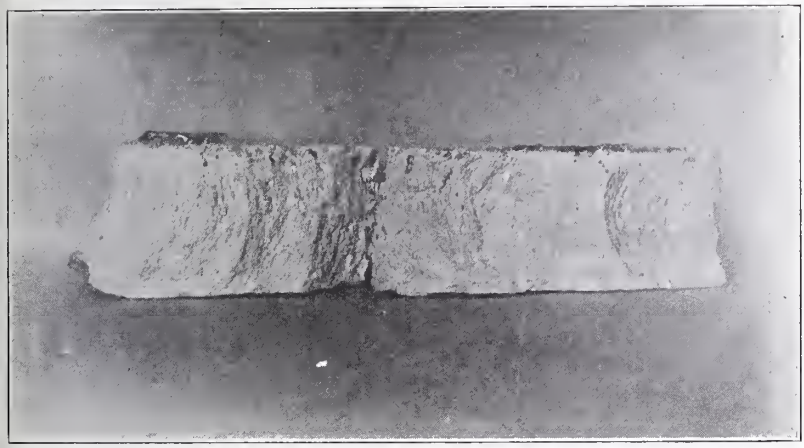

FIG. 3.

the bolts the plank that holds the core is then removed. The core is then lifted out by the crane. This may bind somewhat to the ring or the pot may have shrunk around it but it should release without using force. If the pot has remained in the mold too long it will shrink around the core so tightly as to cause splitting.

The core is placed on the floor directly back of the rest of the mold. It should be placed on a pad of some sort to prevent scratching. The tie board is placed across the top of the core and the ring is lifted by hand off the case and put on top of the board. This leaves the inside and top of the pot exposed. If the pot

is too soft to stand alone, the shell should be left so as to support the pot until stiff. If the core is drawn about 9 A.M., as it should be, the pot will be stiff enough to move at 2 P.M. Sometimes it may be removed at once.

When the pot is of proper stiffness the crane is hooked on to the mold frame-support and the whole arrangement lifted; including frame, shell, bottom and pot. When the bottom of the frame clears the top of the pedestal the frame is turned 90° and the load gradually lowered. When the bottom rests on the pedestal it will release from the shell as the shell continues to lower and the bottom and pot will be left on the pedestal and the frame and shell lowered into the bottom of the trench. The trench can then be covered over with planks, if desired.

To remove the pot from the pedestal a special sling is used as shown on the drawing. This consists of two timbers arranged on collapsing hold-rods which can be put under the plaster bottom, one on each side of the pedestal. By having these rods so that the two timbers can be spread, they can be lowered down over the pot and base while spread. They are then folded in snugly to the sides of the pedestal beneath the base. When the sling is placed, the pot is picked up and lifted over to the runway between the two lines of molds and lowered down on to a suitable skid board for use with a lifting truck or transveyor. The width of the platform must not be any greater than the width of the pedestal and its length must be great enough so that the inside distance between the two skids allows clearance for the truck to be slid under the platform.

In setting the pot and bottom down on the platform, the platform should be placed with the narrow-way crossways to the aisle so that the truck when slid under will be in position to be pulled away without any turning.

The pot is left on the plaster base until stiff enough to move. There should be enough extra mold bottoms provided to meet whatever requirements there are.

In fitting the mold together again for a new cast a dry bottom should be placed on the pedestal and the shell lifted around it.

It is not considered necessary to discuss drying and storage.

There are several modern sytems which may be successfully employed.

Figs. 4 and 5 show a split mold and a pot cast in same. These illustrations show what is actually being done with hooded pots

FIG. 4.

FIG. 5.

of the regular glass-house type. The hood and bottom are cast in one piece up to the line shown in Fig. 5. The top of the hood and cover are cast in a separate piece. When properly stiffened, the top is turned over and stuck on to the bottom. The casting of the two parts separately is necessary on account of the use of the core. Molds for pots of this type can be arranged to work with a shell and inside bottom to the mold, as shown in Fig. 2.

Summary.

Sufficient casting work has been done to demonstrate that this process can be adapted to a great many products as well as glass pots, and that its use is worthy of consideration.

Experiments are being made commercially with pots similar to the one illustrated in Fig. 5. It may be interesting to note that one of these pots has been in use with lime-soda glass for a period of 16 weeks and has already been charged 96 times. The body used is of the type given above.

In conclusion the writer desires to say that the methods described have been developed through coöperation between the Clay Products and the Glass Sections of the Bureau of Standards.

Bureau of Standards,
Pittsburgh, Pa.

NOTE ON THE CASTING OF PORCELAIN GLASS POTS.[1]

By J. W. Wright and D. H. Fuller.

The procedure of casting glass pots has been described by several contributors to This Journal.[2] It is the purpose of the present paper to call attention to several points of practical importance.

The characteristics of a casting slip as well as of the fired body depend, first, upon the composition—the content of grog, kaolin, ball clay, and feldspar; second, the sizing of the grog; third, the peculiar physical and chemical qualities inherent in primary kaolins as differentiated from the secondary ones, and in ball clays against the fire clays; fourth, the amounts and proportions of sodium silicate and carbonate of soda.

The following mixtures have been used successfully in the Pittsburgh Laboratory of the Bureau of Standards for the casting of glass pots:

Mixture No. 1.

	Per cent.
Whiteware-bisque grog	48
Feldspar	7
Kentucky ball clay, No. 4 or No. 6	11
Tennessee ball clay No. 5	11
Georgia, Florida, Delaware and North Carolina kaolins, 1/4 of each	23

Mixture No. 2.

	Per cent.
Whiteware-bisque grog	48.0
Feldspar	7.0
Kentucky ball clay No. 4	8.5
Tennessee ball clay No. 5	8.5
Georgia, Florida, Delaware and North Carolina kaolins, 1/4 each	28.0

[1] By permission of the Director, Bureau of Standards.

[2] A. V. Bleininger, *J. Am. Ceram. Soc.*, 1, 15–24 (1918); F. H. Riddle, this number, pp. 647–658.

Mixture No. 3.

	Per cent.
Whiteware-bisque grog	48.0
Feldspar	6.5
Kentucky ball clay No. 4 or No. 6	11.0
Tennessee ball clay No. 5	11.0
Kaolins, as before	23.5

Mixture No. 3 has been used more extensively than the others. It is evident that the composition, in general, governs the final qualities of the pots, as is true of all ceramic bodies.

The sizing of the grog, in this case the waste bisque obtained from whiteware potteries, is quite an important factor. It is especially desirable to avoid too large a proportion of fines passing the 80-mesh sieve, since they promote vitrification too decidedly. While it is intended that the pot body should be dense and nearly non-absorbent, it is not good practice to develop a glassy structure. This objection would not hold if the grog possessed superior refractoriness and the fine particles by absorption became effective in increasing the rigidity of the mass. The mechanical analysis of the grog used corresponds fairly closely with the following sizing:

	Per cent.
Between the 10- and 20-mesh sieves	30
Between the 20- and 40-mesh sieves	40
Between the 40- and 80-mesh sieves	17
Through the 80-mesh sieve	13
	100

The physical and chemical qualities of the clays used are of evident importance with reference to their response to the effect of the alkaline electrolytes. The primary kaolins are most responsive, followed by the secondary kaolins, ball clays and fire clays in the order given. As a rule, the more plastic the bond clays, the smaller will be the amount of water required in the slip and the quicker the time of casting. Distinct differences were noted in the use of the several ball clays.

Slips have been made containing mixtures of three ball clays, Tennessee No. 5, Kentucky No. 4 and No. 6 in various proportions. The Tennessee No. 5 is generally very plastic and pos-

sesses good bonding properties; it increases the specific gravity of the slip and lowers the water content. At the same time it is effective in reducing the time of casting, lowering the shrinkage and the time of drying. It seems to be affected more profoundly by the reagents used, a fact associated with its fineness of grain and plasticity. The Kentucky ball clay No. 4 has fair plasticity and can be used to replace the Tennessee No. 5 clay more or less completely. It has been used alone to very good advantage but its slightly inferior plasticity necessitates the use of a larger quantity. The Kentucky ball clay No. 6 resembles a fire clay in its properties. Its plasticity and bonding power are lower than those of the preceding materials. In a slip containing some of this material the water content and shrinkage are higher, and the specific gravity lower. The strength likewise is decreased. It may be used because of its property of firing to a dense structure at lower temperatures.

From 17 to 22 per cent of ball clay has been used in our work, the higher amount giving better slips for casting. Definite limits regarding the use of any one ball clay can hardly be given since successive shipments rarely show the same physical properties. Any variation, therefore, might necessitate increasing or decreasing the quantity used. If several ball clays are used a slight change in the properties of one will not upset the working qualities of the mixture.

The presence of soluble salts in clays often causes considerable trouble in that the slips stiffen as soon as agitation ceases. It has been found desirable to add 0.01 per cent of barium carbonate with the alkaline electrolytes for the purpose of eliminating the soluble sulphates.

The use of the sodium carbonate and silicate has for its object reduction of the water content, formation of a dense, heavy slip, decrease of shrinkage, and the development of a capillary structure which makes possible increased speed and safety of drying. The amount of these salts which can be used varies from 0.20 to 0.33 per cent of the anhydrous compounds in terms of the dry weight of the clay and non-plastic materials. With any one composition the relative proportion of the two salts seems to be of greater im-

portance than the total quantity used. The proportion which has given the best results is 0.12 per cent of sodium silicate to 0.09 per cent of sodium carbonate. With this ratio the water content is approximately 22 per cent of the weight of the dry materials. A water content of 25 per cent was found to be required when the salts were added in the ratio of 1 : 1. It has been shown to be desirable not to introduce both salts at the same stage of the blunging. The silicate is dissolved in the water and then the dry body materials, thoroughly mixed, are added. This mixture is blunged to form a fluid slip and the carbonate is then added. The viscosity of the slip is increased somewhat by this addition of electrolyte. The increased "stiffness" of the mass seems to be apparent as a kind of stickiness rather than greater viscosity, due to coagulation, and the slip resembles heavy molasses. A further addition of silicate at this point lowers the viscosity, which indicates that the ratio of the salts is of primary importance.

The high viscosity desired is such that the pot will cast solid over night, in approximately 17 hours. The higher the viscosity, the slower is the casting of the slip, the greater the drying shrinkage, and the longer the time required for drying. With very viscous slips but little absorption of water by the plaster mold takes place—as the mass stiffens too quickly to permit of flow. However, with such a slip the pot does not become solid for some time.

With a slip of low viscosity considerable flow occurs, due to the absorption of water. An undesirable structure may develop when the slip passes down within the partially-stiffened outer portions of the pot wall. The less viscous slips, however, become solid in a shorter time and show a lower drying shrinkage. Control of viscosity must arrive at a compromise between the two extremes.

The time of casting is controlled to a considerable extent by the proportion of the two salts. With slips of practically equal viscosity an increase in the amount of sodium carbonate increases the time required for casting. Several attempts were made to cast pots using the proportion of 2 sodium carbonate to 1 of silicate. Very little or no casting occurred and the pots remained soft for 20

hours or more. They would crack in the mold and yet be too soft to allow the removal of the core. Pulling the latter would cause the pots to soften out of shape. On the other hand, with a proportion of 1 sodium carbonate to 2 of silicate, the slip was exceedingly heavy, with a specific gravity of 1.96, and yet of low viscosity. These slips did not stiffen, when allowed to stand, like those higher in carbonate. They permitted high absorption by the mold, showed low shrinkage, and cast solid in from 11 to 14 hours. The pots cast from these slips possessed a good exterior appearance but their internal structure showed much flow and lamination. The proportion of 4 sodium silicate to 3 of carbonate invariably gave the best results with reference to good casting quality, time of setting, moderate drying shrinkage, and satisfactory strength. The drying shrinkage of such pots is about 1.84 per cent in terms of the dry length and the burning shrinkage 6.82 per cent. The condition of the mold also affects the time of casting and, as is to be expected, the drier the plaster the more rapid is the absorption of water.

Summarizing these points regarding the casting of glass pots, it is apparent that the water content must be low, usually between 22–25 per cent in terms of the dry weight of the materials, the specific gravity of the slip from 1.90 to 1.95, and the viscosity quite high. The body thus produced must have low drying shrinkage and high mechanical strength. The amount of electrolyte varies from 0.20 to 0.33 per cent of the weight of the dry materials and the best ratio of sodium silicate to the carbonate is 4 : 3. An increase in sodium silicate decreases the water content, lowers the viscosity, and shortens the time of casting. An excess of it produces flow or laminated structure.

Kaolins respond most readily to the action of the alkalies, and fire clays least. In slips the use of a more plastic clay in place of a leaner one (fire clay) brings about higher specific gravity, shortening of the time of casting, decreased drying shrinkage, and increased strength. The use of several plastic clays is recommended and the introduction of a small amount of barium carbonate seems desirable.

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Pittsburgh, Pa.

CERAMIC ABSTRACTS.

General.

The osmotic purification of clay. W. R. Ormandy. *Pottery Gazette*, **505**, 705–7 (1919).—The term "endosmosis" is illustrated as follows: If a glass vessel containing water is divided into two compartments by means of a porous diaphragm, and two oppositely charged plates are put in the compartments, it is found that the water will go up on one side of the diaphragm and down on the other. Furthermore, if one placed a battery of capillary tubes of extreme fineness under water and put two electric plates in the same, the liquid would move through the tubes. If the glass tubes were coated with sulphur on the inside the movement would be in the opposite direction. Count Schwerin, in experimenting to drive off water from colloidal peat (containing 95 per cent water), used a wooden screen with fine brass wire gauze. The peat was put in the screen and covered with an iron plate which acted as one electrode while the gauze acted as the other. A current of electricity was passed through same and immediately there was a rain-fall which in 20 minutes reduced the 5-inch layer of peat to $^1/_2$ inch. The layer of peat at the bottom of the screen acted as though it were a series of capillary tubes. Experiments with clay showed that some clays would collect at one of the poles while others would not. Many that would not in the natural state could be made to do so by deflocculation with alkalies. In cases where merely treating with alkalies did not help, the clay was first pugged with humus which aided the clay particles in picking up the sodium and then the clay could easily be collected on the poles. Clays purified and dried by this process are as dry as clays subjected to a pressure of 150 lbs. per sq. in. in a filter press. Drying of the clay is explained by endosmosis. The clay is first deposited on the drum in a thin layer which immediately forms a series of capillary tubes through which the water is driven out. Nearly all clay particles are negative. Silica is neutral and iron hydroxide is positive. By adding the right amount of alkali one group of particles could be neutralized giving off a charge which will excite the other particles. Pyrites can easily be separated from clay. Vitrification points may be lowered as much as 300° and the plasticity is increased. H. G. Schurecht.

Analysis of minerals and alloys of zirconium. A. Travers. *Chimie & industrie*, **2**, 385–92 (1919).—Baddelyte, the principal Zr mineral, contains 75–98% ZrO_2 and as impurities SiO_2, TiO_2, Fe_2O_3, etc. Zircon, $ZrO_2.SiO_2$, contains in addition to the ordinary impurities rare earth phosphates. Before decomposing these minerals must be very carefully pulverized. Satisfactory decomposition cannot be brought about by fusion with KF, $KHSO_4$, Na_2CO_3 or KNO_3. Fusion with Na_2C_2 gives rapid and complete decom-

position. *Analysis of zirconia.* Fuse 1 g. finely powdered mineral with 10 g. Na_2O_2 in a nickel crucible. As this introduces Fe and Mn these must be determined on a separate sample fused in a porcelain crucible. Use separate samples for determination of Zr and Si. *Detn. of ZrO_2.* Treat fused mass with 300–400 cc. water, boil, filter on a large filter and wash with hot water containing 5% carbonate. Wash residue into a beaker and add ash of filter after igniting. Boil with excess HCl, add excess NH_4OH, filter, and wash with 10% NH_4OH solution. Redissolve in hot acid, cool, and neutralize almost completely with NH_4OH. Add excess $Na_2S_2O_3$, about 5 g. in 10% solution, boil till no more SO_2 is given off, filter and wash with hot water. Ppt. after ignition contains ZrO_2, TiO_2, and traces of SiO_2, Fe_2O_3, and Ni_2O_3. Transfer to a platinum crucible, add a few drops of water, 3–4 cc. HF, and 1 cc. H_2SO_4, evaporate to dryness, and calcine at a red heat to constant weight. Fuse residue with 10 g. $KHSO_4$, and dissolve in 10% H_2SO_4. Detn. TiO_2 colorimetrically on an aliquot part and Fe_2O_3 on another part. Detn. ZrO_2 by difference. *Detn. of TiO_2.* Add several cc. H_2O_2 to cold soln. and determine colorimetrically if present only in small amounts. The addition of large excess of H_2O_2 to soln. and boiling will precipitate all the ZrO_2. This can be used as a separation and gives a delicate test for ZrO_2, as 0.0001 g. in 30 cc. can be detected by this reaction. If TiO_2 is present up to 4–5% reduce with Zn and titrate with $FeCl_3$ using KCNS as indicator. *Detn. of SiO_2.* Decompose 1 g. sample as above, dissolve in HCl, evaporate to dryness, take up with HCl, and filter off SiO_2 rendered insoluble. To remove SiO_2 still in solution add excess NH_4OH to precipitate zirconia. Precipitate carries down all silica which is thus removed from presence of sodium salts and can be made insoluble. Dissolve precipitate in dilute HCl, evaporate to dryness, take up with HCl, and filter on same filter. Ignite to constant weight, and smoke off SiO_2 with HF in presence of H_2SO_4 as above. H_2SO_4 is necessary to prevent volatilization of ZrO_2 carried down by SiO_2. *Detn. of Fe_2O_3.* Decompose 1 g. material in porcelain crucible, take up with hot water, filter boiling, and dissolve residue in hot HCl. Boil off Cl set free by MnO_2 and titrate with $TiCl_2$ using KCNS as indicator. *Detn. of MnO_2.* Decompose 1 g. sample in porcelain crucible, take up with hot water, filter boiling, and dissolve oxides in HNO_3. Detn. colorimetrically after oxidation to permanganate by $K_2S_2O_8$ and $AgNO_3$. *Analysis of zircon.* After fusion of very finely ground sample in Na_2O_2 as above take up with water, filter, dissolve residue in HCl, and remove Ni by 2 precipitations with excess NH_4OH. Remove SiO_2 by evaporation in HCl soln., and exactly neutralize filtrate with NH_4OH. Add 5–8 g. pure oxalic acid, boil one-half hour, and allow to stand in cold for 12 hours. Wash precipitate of rare-earth oxalates with 10% oxalic acid in water, filter, ignite, and weigh as rare-earth oxides. Determine ZrO_2 in filtrate as above after recovery of ZrO_2 carried down by silica. This recovery is made by smoking off the silica with HF and H_2SO_4, fusion of residue in $KHSO_4$, dissolving in water, precipitating with NH_4OH and dissolving in HCl. *Detn. of alumina.* When present as in the analysis

of alloys of zirconium, alumina will be precipitated with the zirconia. It may be separated by fusion of the precipitate in pure KOH. After solution in water and filtering alumina is precipitated from the filtrate in neutral solution. The article also contains directions for the analysis of zirconium alloys. J. S. LAIRD.

Geological.

The sand and gravel resources of Missouri. C. L. DAKE. *Missouri Bureau of Mines*, 15, *second series* (1918).—In 1907, O. U. Stromme commenced field mapping of the St. Peter sandstone with a view to getting out a report on the glass sand industry of Missouri. Mr. Stromme was called away from the work which was left until 1912 when it was resumed by Mr. Crane. After a short time he too was called away from the work and since 1913 Mr. Dake has continued the field work. The results of Mr. Dake's investigations which cover several years are embodied in this report which deals not only with the glass sand industry of Missouri but also contains data concerning all other sand and gravel deposits of that state. In this interesting report the general properties, origin and nature of sand are first discussed. The author then proceeds to consider the methods of handling the material and its uses and finally deals with the distribution of sand and gravel by geological formations and districts. In the portion dealing with distribution the various counties of the state are considered somewhat in detail with reference to sand and gravel resources. Numerous maps and illustrations are included in the report. E. D. ELSTON.

High-grade silica materials for glass, refractories and abrasives. R. J. COLONY. *N. Y. State Mus., Bulls.* 203, 204 (1919).—Due to conditions brought about by the war, many imports of refractory materials were either wholly prevented or seriously curtailed. Accordingly, domestic production of materials of this character was greatly stimulated. In the report by Colony, various geologic formations occurring in New York State are cited as being of economic value with reference to refractories and the glass industry. The most important of these formations according to Colony are as follows: The *Poughquag quartzite* in Dutchess and Westchester counties; the *Shawangunk conglomerate* in Ulster and Sullivan counties; *Oriskany sandstone* at Oriskany Falls, Oneida county; the *Potsdam sandstone* forming part of the Paleozoic fringe about the Adirondacks, and also occurring in the Mohawk Valley; the *Oneida glass sands* near Lake Oneida in Oneida and Oswego counties. Colony also states that materials might possibly be derived from the following formations in New York State; the *Whirlpool sandstone* at Lewiston, Niagara Falls, Niagara county; the *Oswego sandstone* at the falls of the Salmon River, in Oswego county; the *Oneida conglomerate* at Washington Mills, Oneida county; and a sandstone underlying the Lowville limestone occurring near Little Falls. The investigations are in general limited to the five formations first mentioned and the report is divided into two parts: (1) The rock and its structure are described briefly and there is

included a more detailed consideration of the lithologic character, chemical analyses and locality for each formation together with suggestions regarding facilities for working and transportation. (2) Tests made during the investigation are stated and mention is made of the various industries in which rock of the different formations might be used. Colony concludes that in the State of New York there are materials which might be used as follows: 1. *For glass making.* (*a*) The Potsdam sandstone of either the Johnstown district or the Moira-Bangor district. (*b*) The Oriskany sandstone, Oriskany Falls. (*c*) The Oneida glass sands. 2. *For ferro-silicon manufacture.* Rock from all the formations cited. 3. *For silica refractories.* (*a*) The Poughquag quartzite, especially a very pure variety occurring in Dutchess county. (*b*) Certain beds of the Shawangunk. 4. *For tube mill liners and pebbles.* (*a*) Especially that pure phase of Poughquag quartzite in Dutchess county. (*b*) Possibly the Poughquag quartzite near Peekskill. (*c*) Possibly certain beds of the Shawangunk. E. D. ELSTON.

Refractories.

Our present knowledge of refractory products. J. BIED. *Ceramique,* [1] **22,** 33 (1919).—Before the war the refractories manufacturers worked mostly on an empirical basis. Today they are improving their methods through technical aid. Already it is safe to predict that France will surpass Germany in this line of manufacture. Great credit is due to the work of H. LeChatelier and his co-worker Bogitch. The present article deals with basic, silicious, and silica-alumina products, little being known in France of the manufacture of the neutral refractories as chrome brick and bauxite. Dolomitic sand is very abundant in France. It is dead-burned in furnaces to a sufficient degree of heat to prevent hydration under ordinary atmospheric conditions for from two to three weeks, a long enough time to allow it to be placed in position for use in a furnace. Pure calcium lime air-slakes at a rate depending on its qualities and may reach temperatures up to about 400° C which is the temperature of dehydration of $Ca(OH)_2$. If the lime grains are large, hydration may be slow. Dead-burned dolomitic lime, however, slakes slowly in air and a year or two after placed in the furnace wall causes much trouble by expansion. Several per cent of iron carbonate or 5 to 6% of kaolin or both materials added together prevent slaking. After being dead-burned the dolomitic sand, or rock, is ground to two sizes, one of flour fineness, the other of size of particles all passing through a sieve of 5 to 6 mm. openings. 30 to 40% of fine-ground and 60 to 70% of coarse-ground material are mixed for making brick or for tamping in place in monolithic construction. The dead-burned material may begin to slake in two weeks, the brick in less time, hence requiring immediate placing for use in a furnace. For monolithic construction the finely-ground, burned dolomite is mixed with tar which has been boiled five hours. The tar with this treatment is very viscous and forms long tough threads which help to hold together the mass of the tar-dolomite mixture. If desired, the mixture may be molded

into brick in a press at a pressure of 400 to 500 kg. per sq. cm. After 4 to 5 hours the brick become quite hard. They must be put in place within 2 to 3 days and burned to a clear red heat. Special forms as those for the bottom of the Bessemer furnace are molded in cast iron forms and the whole placed in reverberatory furnaces and burned to 900° or 1000° C for 8 to 10 days. A converter cover prepared in this manner lasts for from 140 to 150 charges. Before the war all the magnesia used in France came from Styria, Austria, some in bulk form and some as brick. The compositions indicate that artificial cement was added to the magnesite for making brick. The 6 to 8% of Fe_2O_3 in the Styrian magnesite prevents slaking to a great degree but lowers the resistance to deformation at high temperatures. The relation between temperatures and resistance to deformation is shown by a diagram of H. LeChatelier and B. Bogitch (*Compt. rend.*, **165**, 488 (1917) Specimen No. 1. See Ceramic Abstracts in this number). The figures show that the Styrian brick suffers its greatest loss of strength between 1300° and 1400° C. During the war, magnesite has been obtained from Euboea, Greece and made into brick in France. The compositions of the raw materials and of the brick are as follows: No addition to the raw material is made.

MgO	80–85
CaO	4.5–5.5
Fe_2O_3 and Al_2O_3	1.5–2.0
SiO_2	7–8

The degree of calcination is determined from the sp. gr. That of the raw material is 3.92, of the burned 3.4 to 3.5. The calcined product is used in mass construction by mixing with 8–10% of well-boiled tar, the mixture heated to 300° C then pounded into place with tampers heated to dull red heat. For brick two sizes of the burned material are used. The mixture is composed of 2 parts of material less than 5 to 6 mm. in size and 1 part impalpable powder. The relation between crushing strength and temp. of the Grecian brick is shown as Sample No. 2 in the article by LeChatelier and Bogitch already referred to. This brick suffers its greatest loss of strength between the temps. of 1400° and 1550° thus being of much higher resistance than Styrian brick up to 1550°. At this temperature both have practically zero crushing strength. The Grecian brick has the disadvantages of continued shrinkage at prolonged high temps., and ready slaking in air on account of the low amounts of flux. The brick disintegrates in water vapor in 2 to 20 hours. Certain of the commercial brick produced in France were burned in an electric furnace. At the beginning of the burn the porosity was 20%. At the end the porosity was 11% and the volume shrinkage 16%. The porosity should be reduced to a certain minimum but not too low. Italian magnesite is also now used in France. Typical compositions of the dead-burned material and of the brick are given below.

	Dead-burned.	Brick.
SiO_2	8.9	5.15
Al_2O_3	0.27	0.75
Fe_2O_3	1.98	6.00
CaO	4.50	3.37
MgO	85.25	84.00

It is seen that Fe_2O_3 is added for making brick. The Italian magnesite contains many silicious and calcareous concretions which have to be picked out after calcination. This is, however, not a prohibitive handicap for the use of this material. Eight photos are given of plant operations in French factories. F. A. KIRKPATRICK.

Properties of magnesia refractories. H. LECHATELIER AND B. BOGITCH. *Compt. rend.*, **165**, 488 (1917).—6 magnesite and 1 chrome brick were studied. Table 1 describes the brick, Table 2 gives chemical compositions, Table 3 gives crushing strength at different temperatures.

TABLE 1.

Materials Investigated.

No. 1. Styrian brick, burned to 1450° C, made in 1890.
No. 2. Brick of good quality from Euboea, Greece, made in 1910.
No. 3. Brick G of good quality from commercial plant.
No. 4. Brick B, of low quality, containing an addition of 3% calcined pyrites to the mixture.
No. 5. Pure magnesia, melted in the electric furnace and contaminated in the process.
No. 6. The raw material of Brick B, burned in the electric furnace.
No. 7. Chrome brick.

TABLE 2.

Chemical Analyses.

	1.	2.	3.	4.	5.	6.	7.
MgO	86.7	93.4	89.4	81.2	93.7	88.5	12.3
CaO	1.0	3.7	4.5	4.8	2.7	4.5	5.8
Fe_2O_3	6.0	0.5	1.1	4.2	0.3	1.4	15.5 (FeO)
Al_2O_3	0.6	0.2	0.8	1.0	1.1	0.0	10.9
SiO_2	6.7	2.8	4.2	8.8	3.2	6.5	4.7
Cr_2O_3	..	..	..	..	..	..	50.0
Mn_2O_3	..	..	..	..	..	..	1.5

TABLE 3.

Crushing Strength in Kg. per Sq. Cm.

No.	15° C.	1000° C	1300° C.	1500° C.	1600° C.
1	145	85	66	3.6	1.8
2	420	...	..	185	8.0
3	390	...	..	90	4.8
4	230	...	..	16	3.5
5	...	...	..	90	6.6
6	530	...	..	...	3.5
7	260	120	6	2	1.0

Additional values determined for Nos. 1 and 2 may be read approximately as follows from the curves given:

No. 1—800°, 110 kg.; 1200°, 82 kg.; 1340°, 25 kg.; 1200°, 6 kg.
No. 2—1400°, 250 kg.; 1550°, 30 kg.

There was found to be for all the samples a rapid decrease in strength throughout a certain narrow temperature range, the temperature depending upon the purity of the material. The results explain the reason for the better resistance of silica brick in the walls of a furnace, since the silica refractories have higher fusion points, and greater strength at the higher temperatures.

F. A. KIRKPATRICK.

Notes on refractory materials. J. W. MELLOR. *Pottery Gazette*, 503, 493, 494 (1919).—The great bulk of refractories originate from fire clay. The first test to be made in testing fire clays is the cone test because of its cheapness and rapidity. Chemical and rational analyses are desirable. Best results in rational analysis are obtained by calculating same from the ultimate analysis. By calculating the chemical formula with alumina as unity, Ludwig found a relation between refractoriness and composition which was found to agree with nearly 70 British clays—there being only three exceptions. This applies to only the high-grade clays. The heating of alumina causes its specific gravity to increase from 2.8 at 600° C, to 3.8 at 1200° C, with a shrinkage of 30 per cent. Fused bauxite is used in France to make brick under the name "corindite." The presence of ferric oxide acts as a stimulant in converting quartz to a low specific gravity form. Excellent silica brick are made in South Wales with 5% ferric oxide. Dusts of the oxides of zinc, copper, and iron in a reducing atmosphere penetrate rapidly into firebrick and there is evidence of volatilization of metals at unexpected low temperatures; copper at 600° C and iron at 1100°. When coal is salty the volatile salt is active in forming a slag with the clay. Regarding thermal conductivity Mellor agrees that porous materials have a low conductivity at low temperatures but believes that at high temperatures a highly porous structure favors conductivity—due to convection of heat.

H. G. SCHURECHT.

Silica refractories. Factors affecting their quality and methods of testing the raw materials and finished ware. D. W. Ross. Bur. Standards, *Tech. Paper*, 116 (1919).—A review of the literature is given, particularly the recent articles by McDowell, and Nesbitt and Bell. Inversion temps. of quartz, tridymite and cristobalite are discussed. Eutectic points of lime-silica, alumina-silica and lime-iron oxide curves are given. In a review of the geology of quartzites, chalcedony, etc., it is stated that the order of merit for silica refractories is chalcedony, old quartzites and vein quartz. The Medina or Tuscarora formation in Penn. supplies over half the rock used in U. S. for silica refractories. The rock is crushed in a wet-pan and 2% CaO by wt. is added. When thoroughly mixed, the bricks are molded by hand, hot-air dried and burned to cone 14–18. In European countries bricks are made containing 96% silica, about 1.3% or less of lime and a little highly silicious clay. Silica bricks are used in regenerative and coking chambers of by-product coke plants; tops, ends and sometimes, walls of open-hearth furnaces, etc. A number of heating tests were made on raw quartzites, raw commercial brick mixes, commercial brick and special mixes, to determine the amount, speed and nature of vol. changes caused by various heat treatments. The leading varieties of silica brick made in U. S. were subjected to analyses and softening temp. tests, load tests and vol. changes at 300° C, micro studies, slag tests, screen analyses, cold strength tests, and vol. change tests. Voluminous data for all these tests are recorded. Rates of transformation of different materials to cristobalite on heating to 1400° are in the order: amorphous silica, chert, chalcedony and quartz glass. All are transformed at about equal speed and addition of lime hastens transformation while plastic clay has no effect. Due to small percentage of flux, quartzite is transformed to cristobalite in burning but after 70% has been inverted, tridymite makes its appearance and develops slowly with continued heat treatment. Excepting special cases, the sp. gr. of silica brick is between 2.65 and 2.27, and sp. gr. determinations show the degree of burning the brick has had. In burning, the vol. of solid material increases 10–15%. The average brick contains about 27% pore space and the porosity of burned brick is greater than that of the raw mix. Minimum porosity can be obtained by raising the temp. at the usual rate to 1250–1350° and holding for several days. Transformation of quartz will then proceed at moderate rate and the soaking will permit the fluxes to exert maximum effect in drawing the materials together, thus keeping increase in pore space down to a minimum. The temp. should finally be raised to cone 18–20. The chief thermal volume changes in silica brick are due to tridymite inversions below 200° and the alpha-beta eristobalite inversion at 220–300°. Practically all spalling occurs between atmospheric temp. and 300° and silica brick when heated in a load test show marked volume expansion in this temp. range. Strength tests show lack of uniformity. A mix which yields satisfactory results shows the following screen analysis: residue on 8-mesh 15.3%, 16-mesh 13.4%, 20-mesh 4.6%, 30-mesh 5.4%, 40-mesh 4.8%, through 40-mesh 56.5%. Cross

breaking tests indicate that with bricks made from quartzite, the strength of slightly to mediumly burned brick depends upon the state of metamorphosis of the raw material, porosity of the raw brick, and the lime-silica bond formed on burning. Hard-burned brick seem to have their strength increased by the formation, through transformation of quartz, of interlocking crystals. To resist attacks of basic slags, etc., silica brick should have as low porosity as possible and contain as large an amount of large fragments as possible, for it seems that small particles are fluxed away before large particles are attacked. An all-tridymite brick does not seem commercially probable. 6–8% lime increases the speed of the quartz inversion and does not appreciably lower the fusion point. *General characteristics* of best grade silica refractories: (1) Shall contain not less than 95% SiO_2. (2) Fuse not lower than cone 31. (3) Sp. gr. shall not be above 2.384. (4) Permanent exterior vol. expansion at 1500° for $1^1/_2$ hours shall not exceed 6%. (5) Strength tests shall show average modulus of 540 and effective modulus of rupture of approximately 284. (6) Under 25 lbs. load at 1500° for $1^1/_2$ hours, shall not exceed 2% increase or 1% decrease in length.

EARLE N. JESSOP.

Glass.

The attack of pots for glass melting. W. ROSENHAIN. *Engineering, (L) V.*, **107**, pp. 725–7.—The effect of flint, soda-lime and other glasses on small clay pots of purest china clay was studied. Cuts show effect of attack of glasses of different densities. Dangers possible from the use of local protecting devices for pots are also brought out. CHESTER TREISCHEL.

Cement and Lime.

Portland cements. E. CANDLOT. *Chimie & industrie*, **2**, 371–84 (1919).—Recent developments in portland cement manufacture are reported, in connection with processes of burning and grinding. The rotary kiln which is so largely used mainly on account of the high quality of the cement produced is very uneconomical as regards fuel, requiring 25–35% of the weight of cement produced of a good grade of coal. Two types of stationary shaft-furnaces have been developed to a point where they produce cement of quality equal to that produced by the rotary kiln, but with a fuel consumption of only 14–16%. The finely ground raw mix is mixed with the fuel, which may be coke breeze or culm, and compressed into briquettes. The largest kilns of this type produce only 50 tons per day and considerable labor is required in forming the briquettes and discharging the clinker as mechanical processes for emptying the kiln have not yet been perfected. The cost of these operations does not nearly equal the saving in fuel and in power required to rotate the rotary kiln. "Compound" tube mills for grinding the clinker are proving very economical. The largest of this type grinds 10–12 tons an hour and consists of a cylinder divided into two compartments by a cross partition provided with sieves to permit the passage of the rough-ground material

to the finishing compartment from the first or rough-grinding compartment. The latter is half filled with steel slugs, while the finishing compartment is provided with flint pebbles. Calcium-aluminate cements have been made by fusing lime and bauxite together. Such cements cannot be made in the rotary kiln and their hydraulic properties are not destroyed by fusion. They are slow setting (4 hours) but harden very rapidly, 1:3 mortar giving a strength of 450–525 pounds per square inch in 24 hours. Very low-limed cement of composition SiO_2 10%, Al_2O_3 40%, Fe_2O_3 10%, CaO 40% made in this way appear to be very resistant to the action of gypsum and sulphate solutions. J. S. LAIRD.

New plant of the Kelley Island Lime and Transport Company. COMPANY-STAFF ARTICLE. *Concrete Cement Mill Section*, **14**, 49 (1919).—This plant is located at Buffalo, N. Y. and is one of the most modern in the country. There are many special features. Limestone is to be brought by boat from Kelley's Island, Ohio, and Rogers City, Mich., and unloaded at the dock into a storage pile. It will be dried as needed by a rotary dryer fed by an under-fed stoker. All dust is removed by a centrifugal dust collector and sold as asphalt dust. The stone is then ground in Kent Mills and screened to different sizes for agricultural uses. That portion of the material from $^3/_8$ to $1^1/_4$ inches in size of particle is conveyed to a bin above the rotary kiln. This kiln is 8 ft. in diameter and 135 ft. long and requires three hours for the transit of the lime through it. It is fired by gas producer and the waste gases are used for generating 200 h. p. of steam. The burned lime passes through an open bucket elevator into a rotary cooler and into a storage bin. It is sold in bulk or hydrated. For the latter process a Schaffer automatic hydrator is used. The hydrated lime is passed through Raymond mills and dust collectors and elevated to storage bins above the packing machines. No large lump lime will be produced as only small-size material can be burned in the rotary kiln. F. A. KIRKPATRICK.

The cause of unsoundness in cement. B. F. ERDAHL. *Concrete Cement Mill Section*, **14**, 27 (1919).—It is intended to disprove the assumption that excess of free lime causes unsoundness of portland cement. (1) Lime slakes quickly and there is sufficient water present in the pat used for the test. (2) The rotary movement of the kiln causes sufficient mixing to prevent enclosure of free lime in the clinker. (3) A small ball or particle of free lime could not possibly pass through intense grinding without undergoing at least slight fracture which offers opportunity of slaking on addition of water. (4) 2.7 to 16.7% of free lime (CaO) were added to a sound cement. All the mixtures were O. K. in the soundness test. (5) An unsound cement was ground to pass 200-mesh sieve. After one month in an air-tight bottle it passed the soundness test. (6) Dusted clinker was tested. The fused outer shell was sound, the enclosed dust was decidedly unsound. Chemical composition indicated outer shell to be $3CaO.SiO_2$ and $2CaO.SiO_2$. (7) The conclusion is that the cause of unsoundness is under-burning, with the formation and subsequent decomposition of $2CaO.SiO_2$. No discussion is given of the probable effect of MgO or of alkalies on soundness.

F. A. KIRKPATRICK.

CERAMIC PATENTS.*

G. E. Middleton, *Abstractor.*

Glass gathering machine and process. J. O. Hensen and J. Henle. U. S. 1,304,568, May 27, 1919. This invention relates to a machine and process for gathering glass with a punty as distinguished from those machines and processes in which suction means and ladling and flowing devices are employed. The machine is designed so that the punty is manipulated in a manner closely approximating manual manipulation whereby uniform gathers, free from blisters, are obtained, and yet the gathers for each weight of bottle or other article to be produced are said to be of substantially unvarying amount and more uniform as to weight and form than those incident to hand-gathering.

Glass. E. C. Sullivan and W. C. Taylor. U. S. 1,304,623, May 27, 1919. This glass is especially adapted for fabrication into pressed or blown ware intended for culinary or laboratory uses and is said to possess a high coefficient of thermal endurance and high stability, that is, power to resist chemical attack. It is, generally speaking, a boro-silicate having a linear coefficient of expansion less than 0.000004, a thermal conductivity as high as 0.0028, a molecular ratio of silica to base greater than 13 to 1, and a molecular ratio of acid oxide or oxides to basic oxide or oxides of not less than 15 to 1. The following are examples:

	A.	B_1.	B_2.	C.	D.	E.
SiO_2	70	80.6	80.9	90	85	90
B_2O_3	20	13	12.9	6	12.5	5
Na_2O	4	4.4	4.4	3	1.5	...
Al_2O_3	6	2	1.8	1	...	2
Sb_2O_3	...	...	...	...	1	..
Li_2O	...	...	...	...	...	3
	100	100	100	100	100	100

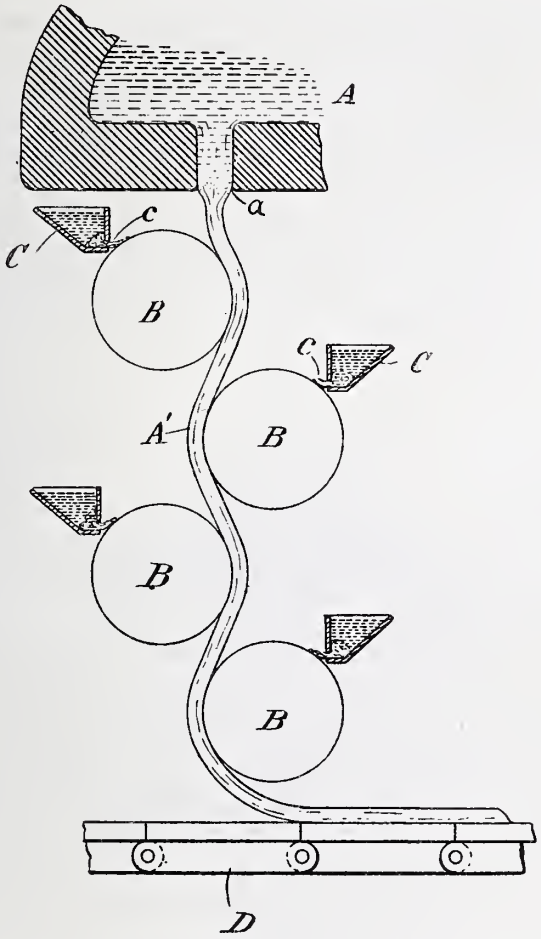
Glass B_2 is said to have gone into extensive commercial use in the manufacture of baking dishes and chemical ware.

U. S. 1,304,622 relates to baking dishes made of this glass.

Process of manufacturing sheet-glass. E. Hopkinson. U. S. 1,305,286, June 3, 1919. Sheet glass is made by causing a stream of molten glass to flow from a source of supply A (see Fig.) free from any contacting surface and then causing it to travel in a sinuous path and thereby effecting surface formation

*Note.—Copies of thsee patents may be secured for five cents each by application to the Commissioner of Patents, Washington, D. C.

on both sides, the concave devices being in contact with forming devices B and the convex surfaces being free during said travel. The forming rolls B



are lubricated at C by means of water or other suitable liquid. Sheet glass made by this process is said to be superior to the ordinary cylinder glass and to approach plate glass in its optical quality.

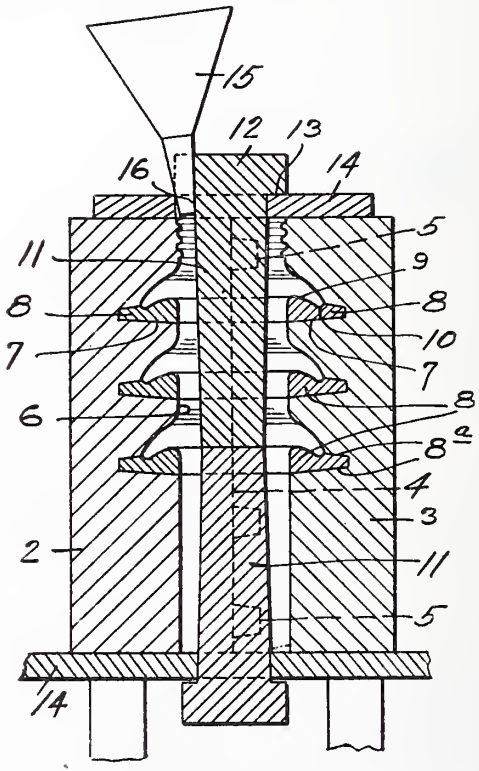
Process and apparatus for drawing sheet-glass. J. H. KENDIG. U. S. 1,305,288, June 3, 1919. Sheet glass is drawn over a horizontally rotating cylinder, the surface of which is of charred bass wood. This cylinder is placed at the delivery end of the tank with its axis at the level of the glass

and by rotating faster than the movement of the glass being drawn, has a polishing action.

Glass. H. P. Gage and W. C. Taylor. U. S. 1,305,793, June 3, 1919. A glass which has high transmission for the ultraviolet rays while absorbing practically all of the visible light. The most desirable results are obtained by a glass of the following composition: SiO_2 50%, K_2O 16%, BaO 25%, NiO 9%. The amount of infra-red transmitted varies with the amount of nickel and the thickness of the glass. If it is desired to reduce the transmission of the red without increasing the percentage of nickel oxide, a small percentage of cupric oxide may be added.

U. S. 1,305,794, is similar to the above but is limited to a zinc glass, an example of such a glass being SiO_2 53%, K_2O 20%, ZnO 15%, NiO 12%.

Mold for forming insulators or other porcelain objects. C. J. Kirk and G. Brain. U. S. 1,304,571, May 27, 1919. This invention relates to the molding of porcelain insulators of "umbrella" type for high tension circuits.



The difficulty of casting the under-cut umbrella sections is overcome by the use of longitudinally split molds 2 and split annular removable cores 8 (Fig.)

Continuous kiln. H. Webster and W. B. Kincaid. U. S. 1,304,831, May 27, 1919. This is a continuous kiln of a modified ring type, consisting of two longitudinal parallel subdivided chambers communicating with each other at either end. The bottom is divided into troughs which communicate with the main draft flues and into which fuel is fed through the crown. The longitudinal chambers are divided into compartments by means of temporary partitions of green brick which terminate short of the crown and, hence, force the products of combustion to pass in proximity thereto. Temporary paper partitions are used in conjunction with the first mentioned partitions in the usual way.

Continuous brick kiln. W. W. Dickinson, Jr. U. S. 1,305,447, June 3. 1919. The permanent flue structure of this kiln is all underground, the kiln itself being temporary and similar to the old scove kiln. It is a continuous kiln of a modified ring type, consisting of two parallel chambers communicating with each other at either end, each of which is divided into several compartments. The gases pass from one compartment to the next downwardly through vertical passages built of burned brick, but filled with green ones. The fuel used is gas or oil.

Manufacture of bricks and furnace-linings from dead-burned magnesite. S. G. McAnally. U. S. 1,305,475, June 3, 1919. Dead-burned magnesite bricks containing 15 to 18% lime, 6 to 6.5% silica, and 8 to 8.5% iron oxide and alumina are made by dead burning magnesite with lime, iron oxide and silica, grinding the resultant product to a powder, mixing with water, molding into shapes and drying and burning at a high temperature. This material is said to be highly refractory, inert to the corrosive action of slags, limestone and molten metal, not to slake with water and to set or bind at a temperature slightly above the normal working of a steel furnace without the addition of other materials.

Diatomaceous composition. T. M. Caven. U. S. 1,305,522, June 3, 1919. This composition is suitable for use in making partitions, roofing, and for insulating purposes, especially where lightness is a desideratum. It is made by grinding diatomaceous material in water to plasticity, burning this product to the desired degree of hardness, crushing it and then adding one part by weight of Portland cement to two parts of the calcined diatomaceous earth and sufficient water

AMERICAN CERAMIC SOCIETY.

The Summer Meeting.

April 4–7, 1919.

The lobby of the Iroquois Hotel, Buffalo, during the forenoon of August fourth, presented a scene familiar to those who attend ceramic meetings. S. C. Linbarger and other members of the local committee had their table in the center and were busy registering the members as they arrived. After registration came lunch and the distribution of tickets for the evening dinner and the Tuesday excursion. The party then took street cars for the Pierce Arrow Motor Car plant. The tour through the factory was rather too hasty to suit the more inquiring but it left time for visits to the Albright Art Gallery and other places of interest. Dinner was served promptly at seven in a private room and about sixty sat down to an excellent repast. Singing popular and familiar songs enlivened the occasion and a short time was devoted to speeches which were very informal, as the evening was to be used for meetings of the professional divisions and the Board of Trustees.

Tuesday was Niagara Falls day. The trip was well arranged by the local committee and the tickets included all charges for the day. The journey was made in three motor omnibuses along the famous brick-paved boulevard, and the city of Niagara Falls was reached about eleven o'clock. The first visit was to the Shredded Wheat plant, noted for its accommodations for visitors, its perfect sanitation, and its wonderful automatic machinery. From this point the party went to the Power Houses and inspected the colossal installations of penstocks, turbines, and generators, by means of which the power of the Falls is harnessed and converted into electrical energy.

Lunch was served on the cafeteria plan at the Chamber of Commerce, after which Mr. Harper, chief engineer of the power company, explained the principles governing the utilization of the Falls and told of schemes of advancement.

The famous Falls were then visited and stops were made at the points of vantage. Crossing the International bridge, the party took a special car to Niagara Glen, through which they were conducted by Mr. Morphy, who has the reputation of knowing every stone and plant in the glen. The varieties of botanical growth were explained and the geological formation was discussed, including the remarkable pot-holes for which the locality is famed.

A sumptuous dinner was served at the Clifton House on the Canadian side, overlooking the Horseshoe falls, and the return journey to Buffalo was made in the cool of the evening.

On Wednesday morning the street cars took the members to the Buffalo Pottery, where they were met by L. H. Bown, general manager, W. J. Rea, superintendent, and other members of the staff. A thorough inspection of the plant was made, special interest being evoked by the electrically driven machinery, the newly installed driers for jigger work and dipping, and the roller machine for underglaze printing. The greatest courtesy was shown by the officials and this proved to be the most profitable and enjoyable visit of the trip.

Next in order was the great Larkin plant in which a great variety of soaps, perfumes, spices, and household supplies are produced or prepared. The automatic machinery proved most interesting and opened up numerous possibilities in view of the present shortage of labor. Before inspecting the plant the party was entertained at luncheon by the Larkin Company and during the meal a motion picture film was shown illustrating the "Larkin idea." Mr. J. D. Larkin, the head and originator of the firm, spoke a few words of welcome to the members.

The huge plant of the Lackawanna Steel Company was next visited and the operations of steel manufacture were followed with interest. The Bessemer converter, always spectacular, delivered its Niagara of sparks and glowing ingots were transformed by the rolls into fiery serpents.

The boat "City of Buffalo" of the C & B Line transported some forty-five of the crowd to Cleveland, over a calm lake illumined by a half-grown moon. Cleveland was reached early Thursday morning and at the Hotel Cleveland an additional number of members was met. A local committee had here made excellent

arrangements and after a welcome breakfast automobiles and street cars transported the visitors, some to the research laboratories of the National Lamp Works at Nela Park, and some to the plants of the Enamel Products Company and the Cleveland Metal Products Company.

At Nela Park the party was met by a guide who explained the interesting historical collection which showed the evolution of the electric lamp in all its parts. They were then conducted to the auditorium where a film was shown which illustrated in great detail the winning of the raw materials and the manufacture and assembling of the modern lamp. It was explained that, as the manufacture was carried on as to the various parts in widely separated parts of the country, it would be manifestly impossible to view the actuality, and in fact the film showed the details in the most thorough manner. At the same time some regret was expressed that the laboratories themselves were not opened for inspection.

About twenty-five persons were driven to the plant of the Enamel Products Company, where Leon J. Frost conducted the party through the factory and explained the details of the manufacturing process. Following this, the plant of the Cleveland Metal Products Company, one of the largest producers of high grade enamel ware in the country, was visited. Before going into this plant it was announced that those going through thereby granted permission for a representative of this company to visit their plants in return. All agreed readily to this proposition. The equipment and grade of ware made in these two plants are fully up to the standard of enameling concerns in this country.

Thursday afternoon was given over to gaiety. The scene was the Cleveland Yacht Club, situated by Rocky River, about ten miles west of the city. The Divisions on Glass and Enamel took advantage of the occasion to hold meetings but these did not interfere with the fun. Several donned bathing suits and disported themselves in the water. Others organized a baseball game in the court with F. G. Lord and F. W. Walker, Jr., as captains. Hits and runs were many and the final score was 19–15. The winning team had some advantage by virtue of being on the Lord side.

Dinner was served, with the compliments of the Northern Ohio Section, and was accompanied by dancing. This was followed by "retreat" by the local Naval Boy Scouts, with the sunset gun and the lowering of the flag. Some of the party then returned to the city, while others continued to dance until a late hour.

The local committees are to be congratulated upon very excellent arrangements and efficient management. The Summer Meetings are necessarily becoming more and more social in their nature, as there is increasing difficulty in obtaining admittance to scientific establishments, but the meeting this year combined both elements in a very happy way.

CHARLES F. BINNS, *Secretary.*

ACTIVITIES OF THE SOCIETY.

Actions taken by the Board of Trustees at a meeting held at the Hotel Iroquois, Buffalo, N. Y., Monday evening, August 4, 1919. Those present were R. T. Stull, Pres., R. H. Minton, Vice-Pres., R. D. Landrum, M. F. Beecher, H. F. Staley, Chas. F. Binns, Secy., Norah W. Binns, Assistant Sec'y.

1. It was voted that, for the present year, 10 per cent of the dues and fees, as of 1919, paid by a member of the Society, who has or shall become a member of a professional division, be allotted to that division for legitimate divisional expenses, such as postage, stenographic services, stationery, printing, and multigraphic work. An accounting of money expended shall be rendered by the Secretary of each division to the Secretary of the Society at the end of the fiscal year.

(NOTE.—In the case of a member who wishes to belong to more than one division, he shall designate his "major" division, and 10 per cent of his dues and fee shall be allotted to that division.)

2. It was voted that the purchase of other volumes in order to obtain those low in stock shall be left to the discretion of the Secretary, it being the sense of the Board that such purchase is wise.

3. It was voted that the list of members of the Society shall not be available to anyone not a member, and that the Year Book shall not be sold separately.

4. It was voted to create a department in the Journal, of $1/2''$ single column advertising, free to members of the Society, for ceramic employers wishing men, and men wishing ceramic positions.

5. It was voted to amplify the booklet on the scope and purpose of the Society and to keep a supply constantly available.

6. Mr. F. H. Riddle was appointed chairman of the Membership Committe in place of Mr. R. C. Purdy, resigned.

AMERICAN CERAMIC SOCIETY.

Acquisition of New Members during July, 1919.

Resident Associate.

Back, Robert, Haeger Potteries, Dundee, Ill.
Crimmel, A. C., Sneath Glass Co., Hartford City, Ind.
Crimmel, H. H., Sneath Glass Co., Hartford City, Ind.
Denk, F. J., 1216 House Bldg., Pittsburgh, Pa.
Fuller, Donald H., Bureau of Standards, Pittsburgh, Pa.
Houk, F. J., Illinois Pacific Glass Co., San Francisco, Cal.
Kimes, A. W., 733 Summerlea St., Pittsburgh, Pa.
Middleton, George E., U. S. Patent Office, Washington, D. C.
Stewart, John G., 530 Union Trust Bldg., Cincinnati, Ohio.
Young, Everett T., Peekskill Fire Brick Works, Peekskill, N. Y.

Foreign Associate.

Murai, S., Ceramic Dept., Central Laboratory, South Manchurian R. R., Dairen, South Manchuria, China.
Page, Howard E., China General Edison Co., Inc., 15 Robinson Rd., Shanghai, China.
Pitt, H. H., Christon Bank, Lesbury, England.
Shanks, Ronald J., Dalmeny Barrhead, Scotland.

NOTICE.

The addresses of the following members of the Society are unknown. Any information sent to the Secretary at Alfred, New York, will be gratefully received.

Kenneth R. Ayer	**Max Dorst**	**G. A. Murray**
Harold A. Best	**Robert W. Earle**	**L. Zach Olsson**
R. E. Brown	**Robert F. Ferguson**	**G. M. Raymond**
L. R. Bucher	**Herman A. Hall**	**Geo. M. Schaulin**
John A. Chase	**A. V. Henry**	**H. H. Sortwell**
Harold E. Clark	**E. A. Maddison**	**Wm. W. Stanley**
May E. Cooke	**Joseph A. Martz**	**Thomas N. Horsley**
Jack M. Dell	**T. C. Moorshead**	**C. E. Ramsden**

JOURNAL
OF THE
AMERICAN CERAMIC SOCIETY

A monthly journal devoted to the arts and sciences related to the silicate industries.

Vol. 2 **September, 1919** **No. 9**

EDITORIALS.

CERAMIC RESEARCH.

Considerable emphasis has been placed of late upon the need of more accurate methods and data for conducting ceramic researches. The value of many of the ceramic researches which have been published in the literature is open to question owing to the fact that the measurements were not accurately made—not through carelessness on the part of the investigator but owing to his lack of accurate measuring instruments and fundamental data in outlining and making the tests.

The results secured in a great many of our investigations have been comparative only and have not been based upon fundamental laws and constants. This applies particularly to investigations of glazes, which have been for the most part empirical. In attempting to apply the results and conclusions drawn from investigations of this kind, we are too often confronted by the fact that statements made and general conclusions drawn are not borne out in similar investigations made under slightly different conditions. Our ceramic bodies, glazes and glasses are complicated in the extreme and working as we are with impure materials it is not strange that the conclusions drawn have often been too sweeping.

Fundamental data as to the properties of ceramic materials are gradually being made available but the remaining fields to be covered are of very large extent. But few of our ceramic laboratories have facilities for the determination of the data required—in fact, many of our investigations fall outside the

field of the technical ceramist and should be conducted by trained physicists in well-equipped physical laboratories. This applies particularly to investigations involving the measurement of coefficients of expansion, thermal conductivity, dielectric strength, etc.

As an illustration of the need of reliable data of this kind, let us consider that property, so vital to many of our ceramic products, "resistance to temperature changes." We know in a general way that this property is dependent to a certain extent on coefficient of expansion, thermal conductivity, mechanical strength, etc. We do not know, however, the relative importance of these properties and, if so, we do not have available sufficient reliable data to develop a product having this property. The only alternative is to make a series of comparative tests, trusting to good fortune that a product having the desired resistance to temperature changes will be secured.

Let us encourage to the utmost the compilation of fundamental data pertaining to ceramic research!

CERAMIC MUSEUM.

The plans which are under way for the establishment of a museum for displaying the historical growth and development of the ceramic industry in the State of New Jersey gives rise to the question whether, in the not too distant future, it will be feasible to establish a national ceramic museum.

In our colleges and universities and in some of our national museums we have fairly creditable ceramic exhibits, but these are not complete by any means and nowhere has an attempt been made to depict the history and development of the many branches of the industry which have become firmly established in this country. A well established national museum would be a source of pride and gratification to the present and future generations of ceramists and of great value in depicting the economic value of this branch of our industries.

ORIGINAL PAPERS AND DISCUSSIONS

THE WHITE CLAY POSSIBILITIES OF PENNSYLVANIA.

By Richard R. Hice.

We need say nothing at this time regarding the importance of what we generally designate as the "White Clay Industry." The demand for the higher-grade clays has resulted in a somewhat extensive reconnaissance of the white-clay deposits of the State of Pennsylvania, and a somewhat detailed investigation of two of the more promising horizons, which will be followed by studies elsewhere.

Disregarding the kaolins of southeastern Pennsylvania, which have been extensively worked in the past, the result of this study shows the occurrence of white clays within the sedimentary rocks at four distinct horizons. The studies have not as yet been sufficiently advanced to determine how closely conditions at these several horizons resemble one another, but it is probable that the conditions are not the same at all of them. The clays at some points have apparently been derived from the decomposition of shales and at other places from sandstones or limestones.

Geological Occurrence.

Clays in Cambrian Formation.—If we examine the geological column in Pennsylvania we will find it fairly well represented by the accompanying section (Fig. 1) taken from the reports of the Second Geological Survey of Pennsylvania. Overlying the Pre-Cambrian rocks, in the southeastern part of the State, occur the Cambrian formations, and in this series is found the Chickies Quartzite. This is a formation occurring in a number of places in southeastern Pennsylvania (Map No. 1) extending from a point on the Maryland line, southwest of Gettysburg, in a general

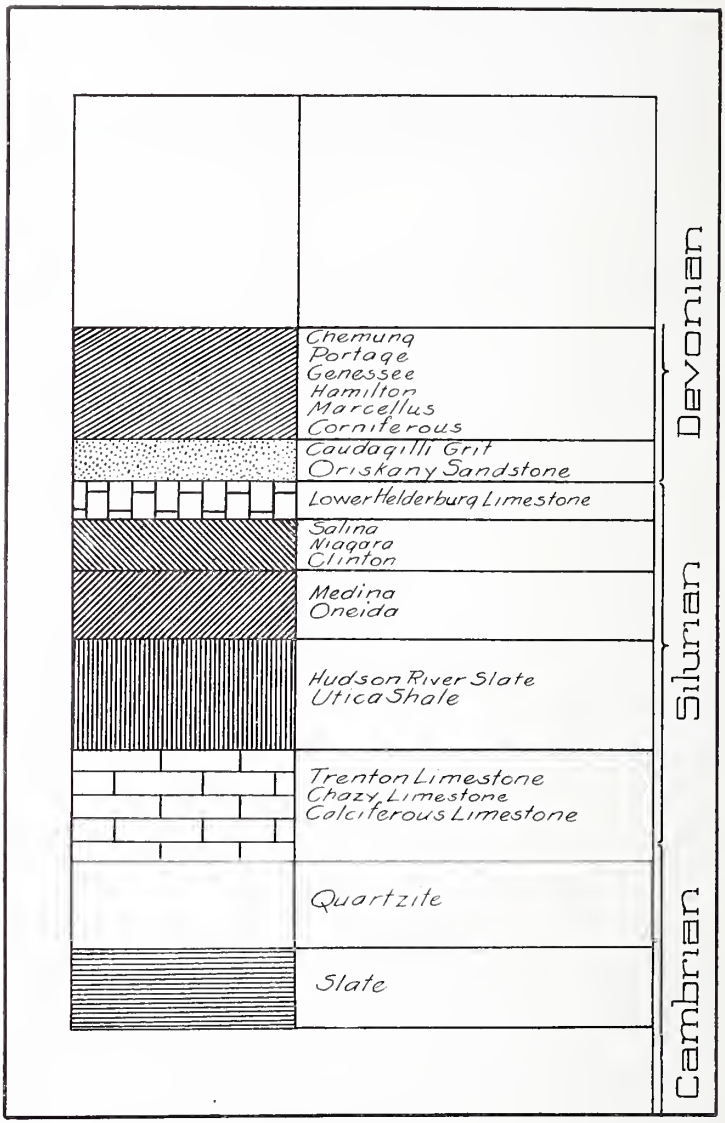
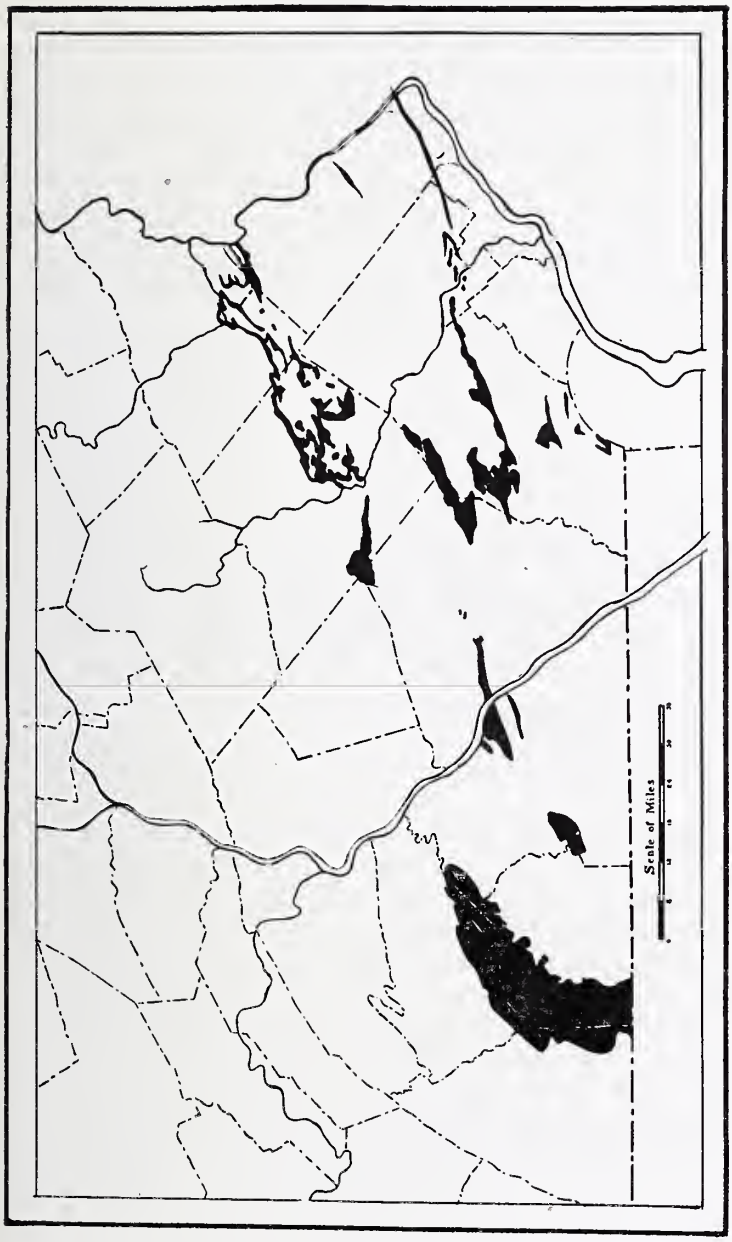

Chemung
Portage
Genessee
Hamilton
Marcellus
Corniferous
Caudagilli Grit
Oriskany Sandstone
Lower Helderburg Limestone
Salina
Niagara
Clinton
Medina
Oneida
Hudson River Slate
Utica Shale
Trenton Limestone
Chazy Limestone
Calciferous Limestone
Quartzite
Slate
Devonian
Silurian
Cambrian

FIG. 1.



MAP NO. 1—Distribution of Cambrian Quartzite in Pennsylvania

northeasterly direction to the Delaware River, a little south of Easton. In this region there are many places where the quartzite is not found, due to the irregular and complicated structure which has been impressed on these old rocks and to erosion which has in many places carried away large areas.

The belt of Cambrian quartzite extends from the Maryland line northeasterly, passing near Mount Holly Springs, through Dillsburg, crossing the Schuylkill River near Reading, touching the Lehigh River near Allentown and Bethlehem, and passing out of the State a few miles south of Easton. West of the Susquehanna River the formation extends as a wide and quite regular mass, while east of Reading it is quite irregular in its occurrence.

Southward from the area described is a second series of exposures reaching from a little southwest of York, through Columbia, where it crosses the Susquehanna River, passing eastward through Honeybrook, Coatesville, Norristown, and reaching the Delaware northwest of Trenton in a narrow band. These occurrences of the Cambrian occur in what may be described as three blocks, the most westerly extending from a point in Lancaster County southwesterly, through Columbia and York; the central area extending from northeastern Lancaster County, near Honeybrook, to a point near Norristown; and the third area extending from Norristown to the Delaware River. Still another occurrence crosses south of Coatesville, adjacent to the Delaware state line. This occurrence is of small extent.

Adjoining the outcrop of the quartzite at many places occurs the limestone series at the base of the Silurian (we are using the names as described and published in the reports of the Second Geological Survey of Pennsylvania) and the clay seems to occur at or near the contact of the quartzite with the overlying limestones.

The best known occurrence of clays at this horizon is, perhaps, in the Mt. Holly Springs region, in Cumberland County, and several descriptions have been published on the clays there. There are two distinct localities near Mt. Holly. The first is a little southwest of Mt. Holly on a small stream known as Furnace Run. For several miles along this stream the overlying limestone occurs, and it is along this portion of the valley that the white clays are found. The clay is quite closely associated with the

iron ores which have been mined in the past in this area. A second occurrence of white clay, near Mt. Holly, is east of the town, also at or close to the point of contact of the limestone and quartzite. It is also being mined at this locality. West of Mt. Holly, at the same general horizon, white clays are reported, but as yet have not been investigated.

Another occurrence of white clay in this same general region is west of Dillsburg. The maps of the Second Geological Survey do not show the limestones as being found here in the same valley as the clay. However, the clays are also closely associated with the iron ores and the old iron-ore mines, and probably the limestones are present. At this point the clay has been opened but was not being shipped at the time of our visit.

Passing northeasterly to and beyond Reading, but very little is known of the clay at this horizon, as it has not been examined as yet.

In northeastern Lancaster County, at Narvon, near Honeybrook, quite extensive clay deposits occur at the same general horizon. Here the clay has a very extensive development, and while not apparently of as good quality as the clay in the Mt. Holly region, it is quite extensively mined.

Clay is also known to occur at this same general horizon in the neighborhood of Coatesville, and northeast, in the neighborhood of Frazer, where, from the little knowledge we now have, it is apparently of much better quality than near Narvon.

A reconnaissance survey was made last summer over part of this region in company with Dr. H. Ries and Dr. F. B. Peck. The finding of clays at a number of points at this horizon rather indicates that they will be found at other places, and considering the relatively high grade of the deposits now known, it may at least be expected that at some points clays will be found of a better quality. Since making the survey of last summer, Dr. Ries reports the receipt of a sample of clay from one point which developed a good color when fired at cone 10.

It is expected that a somewhat detailed study of at least a portion of the possibilities suggested will be made during the coming summer.

Clays in the Gatesburg Sandstone.—Passing higher into the Cambrian formations, we find in Huntingdon and adjoining counties, in the neighborhood of Dungarvin and Warriors Mark, clays in what is designated Cambrian limestone on our old maps. In reality, these clays are associated with what is known as the Gatesburg sandstone. So far as now known these clays are, at least in part, closely associated with the iron-ore pits of that region. They have been studied in some detail by Dr. E. S. Moore for the Pennsylvania Geological Survey and the horizon is well worthy of further examination in other portions of the State where it occurs.

Clays at the Base and Top of the Oriskany Sandstone.—Passing upward in the geological column we come to the base of the Oriskany sandstone—the formation which is so extensively used in Pennsylvania for the manufacture of glass sand. The map (No. 2) represents the extent of the Oriskany in Pennsylvania. This sandstone, while originally deposited as an horizontal formation, has been intensely folded in the process of mountain-making in the State. In general it extends from a point on the Delaware River, a little north of the Delaware Water Gap, in a long and generally narrow belt, southwesterly through Saylorsburg, Kunkletown, Bowmansville, and a little south of Pottsville. It is found then west of the Susquehanna River, south of New Bloomfield, and from there extends in a very irregular exposure to the Maryland state line. Its occurrence is very complicated from the fact that the forming of the mountains brought the Oriskany to the surface in a number of distinct exposures, as is illustrated on the accompanying map (No. 21, there being eleven distinct occurrences of the Oriskany along the Maryland state line. Generally, owing to the character of the Oriskany and the accompanying rocks, it is found in a somewhat vertical exposure. Underlying the Oriskany occurs the Helderberg limestone, and in portions of the State it is separated from the limestone by the thin formation known as the Stormville shales, or the Oriskany shales.

In that portion of the Oriskany exposures extending from the Delaware Water Gap westward through Saylorsburg and Kunkletown to Bowmansville on the Lehigh River, just north of the Le-

high Water Gap, and probably a little west of that point, quite extensive deposits of white clay have been found at the base of the Oriskany. For sometime these clays were thought to have been formed from the decomposition and breaking down of the lower portions of the Oriskany sandstone. Dr. Peck has shown that these clays carry fossils of distinct Helderbergian type, and we must therefore ascribe these clays to the decomposition and breaking down of the Stormville shales, which underlie the Oriskany, and perhaps a phase of the Helderberg limestone. These clays, as has been shown by the mining operations, rest directly upon a sandstone, which the general geology of the region shows is the Shawangunk. The clays at this horizon are being extensively mined at Saylorsburg, and northeast and southwest of that town.

At Shirleysburg, near Orbisonia, in Huntingdon County, a white clay is being mined which is apparently at the *top* of the Oriskany sandstone. This horizon has not been thoroughly examined and nothing is known of the extent or possible occurrence of clays at this horizon, although it is probable that, where the proper conditions exist, clays may be found.

Methods of Mining.

The methods of mining in use vary from place to place. In the neighborhood of Mt. Holly most of the mining is done by drift, which in clays of this character requires quite heavy timbering, and indeed the drifts can be maintained but a relatively short time, as the timbering will not carry the load which is gradually thrust upon it by the slowly creeping clay. At one point, near Mt. Holly, the clay is mined by open cut. The developments near Dillsburg have been made by drifts, while the mining near Narvon is by open cut, there being at that point at the present time an exposure of some eighty feet, with probably forty feet of clay underlying the track level at the present face.

The clays at Warriors Mark and Dungarvin have been mined by open cut, some in the old iron-ore pits, although at places the clay has been directly opened up and is apparently not in contact with the iron ores.

In the region of Saylorsburg the mining is by shaft, and here also

the difficulty of maintaining the workings by timbering is evident. The clay at Shirleysburg is mined by open cut. West of Saylorsburg, near Kunkletown, it is probable that the clay might be mined by open cut.

The proper study of the methods of mining now in use would probably result in changes and lessen the cost of operations. In at least one of the clay mines candles are still used for lighting. At least a portion of the South Mountain area might be mined by open cut, and a thorough study of the methods used would certainly be of advantage to the producers. Apparently not more than 40 per cent of the clay is recovered and the average is probably less than this amount.

Preparation and Uses.

A very considerable portion of the clay now mined is sold in the crude form, no attempt being made to mill it. In general the milling of the clay consists of blunging and screening, with various arrangements of settling troughs and tanks, pressing, drying and pulverizing for the market. Some of the plants use an electrolyte for deflocculation, and acid for again flocculating the clay. In other plants more labor is used than is necessary, due in part to the methods employed. Some plants are not well balanced in their machinery equipment, and were designed apparently without a full knowledge of the behavior of clays and the differences between them and other materials which are ground wet and then concentrated. Without doubt there is room for improvement in the methods of milling in use, and a very marked improvement can probably be made. There does not seem to be a true appreciation of the fact that a very close uniformity of product is necessarily required if the clay is to be marketed as a high-grade material.

The clays as produced are sold both in the raw state and milled. Much of it is used in the steel plants for molding purposes, and some is used in paper manufacture. For the latter purpose it is mainly used as a filler, for most of the clays could not be used for surfacing, which requires a better and more uniform milling to secure a clay which can be used in competition with the imported paper clays.

One of the clays, mixed with ground quartzite or ganister, is used for mortar in the laying of silica brick. Little, if any, is used in pottery bodies. In connection with the possible use of these clays for pottery purposes, it must be said that some appear promising, and it is probable that with better milling they may be used in at least some classes of pottery. These clays carry quite a high per cent of fluxes, and, if so used, provisions must be made for these fluxes in the composition of the batch. Many of the clays carry a high portion of fine silica which should be reduced in amount to allow of their use for ceramic purposes.

Prospecting.

One of the important features in connection with these clays is to know where to look for them. The work done last summer (1918), together with information available from other sources, seems to indicate that it is needless to look for such deposits except at the horizons suggested, and a knowledge of these certainly limits and restricts the areas which are worth investigation. It is useless for us to search for such materials in those portions of the State underlain by heavy horizontal limestones, and it is equally useless to look for good materials where the underlying rocks are heavy, horizontally-bedded sandstones. It would probably also be quite useless in any case to seek such materials where the structure of the rocks, that is, their relation to horizontality, is not suited to the prompt and easy carrying away of the impurities dissolved out in the processes of weathering, as where the rocks have always been practically horizontal. It is for these and associated reasons that the real prospecting for clays is a geological problem.

STATE GEOLOGIST,
TOPOGRAPHIC AND GEOLOGIC COMMISSION OF PENNSYLVANIA,
BEAVER, PA.

COMMUNICATED DISCUSSION.

H. RIES: Dr. Hice's paper calls attention to a series of clay-bearing formations which are of extremely interesting character, *viz.*, the Cambrian and Oriskany. In their unaltered condition they carry little of value, but where the formations carry shales,

schists, clayey limestones or feldspathic sandstones, these are capable of yielding residual clays on weathering, especially, if, as Dr. Hice points out, the beds are tilted so that the surface waters can enter and decompose them.

In Pennsylvania, of course, these residual clays are not in most cases new discoveries, although some of the deposits have been recently opened.

But the point which I desire to emphasize in this connection is that the Oriskany and Cambrian formations which yield these clays in Pennsylvania extend southward along the ranges of the Appalachian province, and that they should be followed, as can be easily done by the use of available geological maps, and prospected for further deposits.

We know that the Cambrian along the west slopes of the Blue Ridge carries a number of white residual clays derived from shales, and the Oriskany has been found to contain white residual clays in West Virginia and northwestern Virginia.

So the search should be continued. As in Pennsylvania, so in the other states where they have been thus far found, the clays are frequently near brown-iron deposits.

Descriptions of many of these deposits will be given in a forthcoming report of the United States Geological Survey.

Cornell University,
Ithaca, N. Y.

APPLICATIONS OF THE POLARIZING MICROSCOPE IN CERAMICS.[1]

By Albert B. Peck

In the rapid advancement and expansion which has characterized industry during recent years and particularly during the stress of the recent war there has been a constant demand for new instruments and devices for research. It has, however, often been easier and more satisfactory to use and develop older instruments along new lines. This is true of the polarizing microscope and, of late, technical practice has turned more and more to it as an aid in solving some of its problems. Sometimes it has been employed alone but very often it has worked hand in hand with the chemist in these tasks with great advantage to both, attaining results which otherwise would have been almost, if not quite, impossible.

While the succeeding pages will deal with ceramic problems, this field is not the only one in which the polarizing microscope is being applied satisfactorily. In a recent article[2] it has been pointed out how the polarizing microscope may be applied to the study of the homogeneity of sugars, salt, flour, photographic developing powders, and chemical preparations and precipitates, thereby detecting adulterants and impurities. In the field of pharmacy excellent results have been obtained recently in applying petrographic methods to the examination of compounds to determine quickly what drugs are present and the purity of other compounds.[3] Perhaps the most recent field into which the polarizing microscope has entered is that of dyes, where its use is contemplated in partially controlling the processes of manufacture of the intermediates and finished products.

[1] By permission of the Director, Bureau of Standards.

[2] F. E. Wright, *J. Am. Chem. Soc.*, **38**, 1647 (1916).

[3] E. T. Wherry, *Ibid.*, **40**, 1063–74, 1852–58 (1918); U. S. Dept. Agr., *Bull.* **679** (1918).

The polarizing microscope[1] is applicable to the study of all minerals or solid substances which will transmit light when in thin fragments and workers in the ceramic field are fortunate in that they make use to such a large extent of inorganic compounds which occur as natural minerals. By the very nature of the development of the polarizing microscope these have received intense study and as a result very complete data concerning their optical properties are available. In the few cases where the compounds encountered in the finished ware cannot be referred to some natural mineral, the very excellent work done by the Geophysical Laboratory in its syntheses often fills the gap. In the organic field, however, the ground has not been so thoroughly covered in the past and this handicap must be overcome in most cases by the production and study of the compounds by each investigator as he proceeds.

When the microscope is spoken of, one immediately thinks of an instrument such as the biological microscope, the chief purpose of which is the examination of minute objects and structures. Such a microscope uses ordinary daylight or artificial light, which is of such a character that its vibrations take place in all directions perpendicular to the direction of propagation. The polarizing microscope possesses all the powers of magnification of the ordinary biological microscope but differs from it essentially in that it makes use of a so-called nicol prism to produce plane polarized light; namely, light vibrating in only one plane. Hence, the name, polarizing microscope.

The earlier polarizing microscopes were used wholly in the study of minerals as they occurred singly or as constituents of the rocks and it is because of this fact that the name mineralogical- or petrographic-microscope is also applied to it. In its early days the observations made with the petrographic microscope were

[1] For complete descriptions of the polarizing microscope and its development, the production of polarized light, and discussions of the various optical properties of minerals in detail, reference may be had to any standard work on petrographic methods such as Idding's "Rock Minerals," Wright's "Methods of Petrographic-Microscopic Research," Johannsen's "Manual of Petrographic Methods," or to F. E. Wright, *J. Am. Chem. Soc.*, **38**, 1647 (1916), or E. T. Wherry, U. S. Dept. Agr., *Bull.* **679** (1918).

almost entirely qualitative in character. It is only in more recent times that it has been used in a quantitative way. While the models of today are essentially similar to the older ones, various improvements in construction have continually been made and accessories added until, in the hands of one trained in its use, it has now developed into an instrument of high precision, both qualitatively and quantitatively.

In carrying out these measurements two methods are in general use. The first is that developed in 1850 by Sorby, an Englishman, the use of thin sections. This method has been the standard method of petrographers and mineralogists. Within recent years another method has been worked out, chiefly through the efforts of the Geophysical Laboratory at Washington in its extensive study of silicate melts. Each has its advantages. The former is most useful in preserving the structure of the body. The latter method, used by the Geophysical Laboratory, is commonly known as that of "immersed grains." The substance is simply crushed to a fine powder, immersed in a liquid of known index of refraction, and the various properties may then be determined on the fragments. It possesses the advantage of allowing the grains to be rolled over and moved around and their optical constants determined in different directions on the same fragment. It furthermore does away with difficulties arising from overlapping grains such as frequently happens in thin sections of fine-grained preparations, giving anomalous results in some cases. The main disadvantage is the loss of structure and texture which sometimes, but not always, results but its advantages seem to outweigh its disadvantages, particularly in fine-grained bodies. European petrographers, however, have not been so quick to adopt it.

Qualitatively, one may study such properties as form, color, cleavage, fracture, vibration directions, and pleochroism. Some of these are self-explanatory but it is not the purpose to discuss them here. Suffice it to say that while they have definite bearing on the study and determination of substances, their importance is not nearly as great as the quantitative measurements.

The four most important quantitative measurements are the index of refraction, double refraction, size of the optic angle, and

extinction directions and angles. Aside from these there are methods of measuring the size of grains in an aggregate and of estimating with fair accuracy the relative proportions in which they are present. The latter, however, has not yet reached the stage where it is as reliable as the other values which may be obtained. Of the quantitative values the index of refraction is by far the most important and the one most often determined. For this reason it may be well to explain how the index of refraction may be determined and used in the study of ceramic materials.

The index of refraction is an expression of the relative velocity with which light travels in air as compared to its velocity in a substance, and by microscopic methods it may be determined with a limit of error of from 0.001 to 0.003 more or less than the true value, under proper conditions and in monochromatic light. In the case of substances such as glass or common salt, belonging to the isotropic group, there is only one index of refraction in all directions but in the anisotropic group to which quartz and feldspar belong, there may be two or three different principal indices of refraction in any one fragment, depending upon what direction the light is traveling through the mineral, and the index for each direction must be determined separately.

The method most used when working with the microscope is that of "immersed grains," previously referred to. A small amount of the finely-crushed material is placed on a microscope slide and immersed in a drop of liquid of known index of refraction,[1] the value of which has been previously determined, and covered with a cover glass. The next step is to adjust the microscope properly so that it allows parallel polarized light to come through and by an iris diaphragm to cut down this light so that only a small amount passes. When we now focus sharply on a fragment we note a bright band of light following the outline of the fragment at its junction with the liquid. This is known as the Becke line effect and by the movement of this bright line as the tube of the microscope is raised or lowered it is possible to tell whether the fragment has a higher or lower index than the

[1] The best practice is to have a set of standardized liquids, ranging from 1.46 to 1.78 or 1.80, which are kept in glass stoppered, covered bottles and differing from each other in index by 0.01 or 0.005.

liquid. For example, suppose the bright line moves into the liquid when the microscope tube is raised. We then know that the liquid is of higher index than the fragment and we take a fresh sample of the powder and immerse it in another liquid of lower index and again note the movement of the Becke line. By using fresh samples of powder and different liquids it is possible to arrive at a point where the fragment is higher than one liquid and lower in index than the liquid next highest in the scale. During this procedure it is also noticed that as the index of the various liquids approaches that of the fragments, the Becke line becomes fainter in intensity and the outline of the fragment tends to merge into the liquid, making it difficult to distinguish the fragment from the liquid. When such a point is arrived at, where the index of the fragment lies between two adjacent liquids in the scale, monochromatic light may be resorted to and the two liquids mixed in various proportions until there is practically no Becke-line effect and the fragment is invisible. The index of the liquid which produces this effect may then be determined on a refractometer and this value then represents the index of refraction of the fragment, with a certain limit of error depending upon conditions which must be judged by the observer.

An alternate method is that of Schroeder van der Kolk. The procedure as far as the preparation of the samples and liquid is the same. Adjustment is made, however, so that converging light is used in place of parallel light and the light is allowed to come through at full intensity. Now when half of the light is shut off by placing the finger or a straight-edged piece of cardboard across the lower end of the condensing system of the microscope, the borders of the fragment appear dark on one side and light on the opposite side. Whether the dark border is toward the shadow of the finger as seen in the field of the microscope or is away from it, determines whether the index of the fragment is higher or lower than the liquid and the observer then knows in what direction to proceed. The change from one method to the other is very easily and quickly accomplished and the two are usually used together; thus one furnishes a check on the other.

A common example of the use of the index of refraction is in the examination of ground feldspar, which is suspected

of having excessive amounts of albite or quartz present because of difficulties from crazing of the glaze. In ground feldspar we commonly find four constituents, orthoclase (potash feldspar) which has indices ranging from 1.519–1.526, microcline (potash feldspar) 1.522–1.529, albite (soda feldspar) 1.532–1.540, and quartz 1.544–1.553. If some of the ground feldspar is immersed in a liquid with an index of refraction of 1.53 and the Becke test applied, we find that the great majority of the fragments have an index slightly less than the liquid, no matter in what direction they may be turned. Many of them show rectangular outlines due to a very excellent cleavage. This indicates that most of the material is orthoclase. Some of the fragments, however, while showing an index less than the liquid in some directions, disappear almost entirely in it in other directions. This indicates microcline. Other fragments which also disappear in the liquid when lying in one direction appear with an index higher than the liquid when turned in another direction. This, then, indicates albite. The fourth constituent shows a higher index than the liquid and also a greater relief or contrast than the albite and this indicates quartz. These facts can also be verified by using liquids of other indices (1.52, 1.54, 1.55) and a check obtained on the previous conclusions. The above procedure can in turn be supplemented by study under crossed nicols, where we find that what was determined as orthoclase shows none or only occasional twinning structures while the microcline shows a very complicated and characteristic fine twinning in which the various sets cross each other at right angles. Albite also shows twinning but it is more simple than that of microcline, while the constituent with the highest index of all (quartz) shows no twinning. Many other phenomena may be used to verify the previous conclusions and with practice one may arrive at some estimate of the relative amounts present and determine whether the albite and quartz are present in amounts exceeding the limit of safety.

The use of a petrographic microscope enables one to gain many advantages. The fineness of the material to be examined will not ordinarily interfere in petrographic work because with the best of modern microscopes practically all the optical properties may be determined on grains as small as 0.01–0.03 millimeter

(1/2500 to 1/1000 inch) in diameter. Some idea of the small size of a grain 0.01 mm. in diameter may be obtained from the fact that such particles when composed of substances like quartz or feldspar, settle as a dust in air at a rate of 25 centimeters per minute and in water at a rate of only 30 centimeters per hour.

Very few branches of science can be said to be really independent of any other and so it is with a petrographic microscope, that while it is often very helpful alone, it is still more valuable when used in connection with the work of the chemist. If the latter wishes to secure pure material, the petrographic microscope enables him to obtain it by detecting impurities and lack of homogeneity. Chemical analysis alone will not enable one to distinguish between a mixture of two substances and a solid solution of the same total chemical composition, except where there is a definite difference in solubilities. With the microscope it is easy to separate the two, since in one case two substances with different optical properties are present and in the other but one substance is seen. Moreover, the optical properties of solid solutions, particularly the indices of refraction, often vary between the limits set by the two end members of the series. This is exhibited by many mineral groups such as the soda-lime feldspars and the olivine group. In the case of feldspars it is possible to determine their chemical compositions with remarkable accuracy by means of the indices of refraction. In Portland cement the amount of free lime or even its presence cannot be detected for a certainty by chemical means alone, while the microscope will readily find this undesirable ingredient by one of several methods.

Another factor which may be of decided advantage at times is the small amount of material with which the petrographer can work when necessary. The average sample of a powder needed at any time when placed on the slide under the objective is hardly larger than a small pin-head and as a general rule not many of these samples are required to obtain the optical properties. Larger amounts at one time make for an undesirable crowding together of the particles in the field of the microscope. It can be seen, then, that the amount of material should cause no disadvantage when only a little is available.

As previously pointed out, the state of the material is not a

matter of great consequence. If it comes to hand in the form of one solid piece, and texture is the principal item of interest, a thin section may be prepared from it. If it is desired to have it in a powdered form, it may be crushed and with the ability to work on grains down to 0.01 mm. in diameter the crushing is not likely to be carried too far.

The saving of time is always an important factor. An instance of the use of the petrographic microscope in this way has recently been given by Dr. F. E. Wright, of the Geophysical Laboratory, in examining some scouring powders.[1] These are usually composed of an abrasive such as ground quartz, flint, feldspar, pumice, or obsidian, and a soap and an alkali such as Na_2CO_3. To make a chemical analysis of such a powder requires considerable time but it is not a difficult matter to distinguish between these three constituents under the microscope. Dr. Wright gave a table showing the relative amounts of each of the main constituents, naming each as quartz, feldspar, etc., soap, or Na_2CO_3, of eight common powders. The whole time consumed was about two hours and the proportions given were of the same order as the longer chemical analysis would subsequently show. No claim is made for absolutely accurate results but the agreement is close enough to give a good indication of their compositions.

The examination of ground feldspar mentioned is another example of how the microscope may be employed to save time. Chemical analysis of feldspar is a matter of days whereas with the microscope a fair idea may be obtained in a comparatively few minutes.

In the following pages some examples from different branches of ceramics will be given in an attempt to show how the petrographic microscope has been used in the past in research to help in the solution of problems. No attempt will be made to include all work along this line nor to give more than brief descriptions of those mentioned. Some are from the writer's own experience while others are taken from widely different sources.

The mineral constitution of Portland cement and the processes taking place in the hydration and setting of it have long created discussion. Many attempts by chemical and microscopic means

[1] *J. Am. Chem. Soc.*, 38, 1647 (1916).

had been made to determine definitely what compounds existed in the clinker but these were without real success until the Geophysical Laboratory, as a result of their physical-chemical and petrographic studies of melts of mixtures of CaO, Al_2O_3, and SiO_2 ventured the prediction of its constitution. Later work by the Bureau of Standards confirmed this prediction. With the constitution of cement clinker definitely settled, the next step was to prepare the pure components and to determine by microscopic study what products resulted upon the hydration of the cement, what components were affected by the hydration, and what, if any, were inert or affected only very slowly and it was found possible to do these things also. From Portland cement the study has been carried on to other compounds of CaO and Al_2O_3, or CaO, Al_2O_3 and SiO_2, which possess hydraulic and cementing properties. In this study of cement the rate of hydration and the change or growth of the crystalline hydration products have been traced microscopically for long periods in molded specimens of neat and sand briquettes and concrete.

The study of porcelain probably represents one of the oldest attempts to use the polarizing microscope along ceramic lines. Once begun, it has been increasingly used to study the changes which take place during burning, to follow the progress of vitrification and the solution of the clay and quartz. The effect on the microstructure of different temperatures of burning has been studied extensively and also the effect of varying lengths of burn. The relation of the sillimanite which is formed during firing to the quality of the ware has also been the object of investigations. The introduction of small amounts of oxides as additional fluxes has been found to have a very marked effect on the microstructure which is reflected directly in the transparency of the body.

Comparatively little work has been done in the microscopic study of glazes. In the case of non-crystalline glazes its use is mainly to detect impurities such as quartz and albite in the ground feldspar leading to crazing, as was mentioned in the discussion of the index of refraction. In the production of crystalline glazes it is very useful in determining what mineral forms the crystals. The zinc orthosilicate, willemite, is the one most commonly formed, although cristobalite, one of the many crystalline forms

of SiO_2, has also been identified. Manganese silicates and oxides have been partially identified and zinc titanate or titanium dioxide crystals are also thought to have been present in some glazes containing those oxides. This field has been investigated so little, however, that it should present possibilities of excellent results.

While the manufacture of window glass and bottle glass has long been established in this country, that of optical glass has only been developed on a commercial scale since about 1917. One of the first things found necessary was the development of suitable pots for the melting of the ingredients. In this, the microscope had a small share in studying the solution effects of the glass on the pot walls. In the earlier stages of production, before sufficient experience had been gained and schedules standardized, it was found quite profitable to follow the melting process by index of refraction determinations with the microscope. These were found to be sufficiently accurate to enable one to tell when the index was near that of the calculated value for the batch and to be sure that the lead, for example, had been raised from the bottom and thoroughly mixed through the melt in the stirring process. Another important result of microscopic study has been not only the determination of the mineral composition of stones in glass but also it has been shown possible to point to the source of the stones, either batch, pot, crown drops, or devitrification. Having found the cause of the trouble, proper measures may be taken to remedy it.

The microscopic study of refractory brick has shown considerable activity in recent years and the investigations have taken in practically all types, silica brick, magnesite brick, various fire brick, brick made from shale and dolomite, and many types of special refractories. All steps from the raw material through burning to the finished product and even on to the product after it had been in use for some time, have been considered. The microscope offers a convenient means of following the mineralogical changes through different ranges of time and temperature in the firing process.

Silica brick in particular have come in for detailed studies and many facts concerning the changes taking place in firing are now

known. For example, it can be seen that the first changes occur in the fine-grained material which acts as a cement in the green brick. In firing, the original quartz inverts to cristobalite which later reverts to the tridymite form which is more stable at the temperatures at which the brick are ordinarily used. After the fine-grained material, the larger grains of quartz shatter and invert to cristobalite along the cracks and in time may be wholly converted to cristobalite followed by tridymite. It has also been found that brick made from a chert, in place of quartzite as the raw material, will invert more quickly to cristobalite and tridymite—probably due to the finer-grained texture of the chert. Much quartz in the finished brick is undesirable since it renders the brick liable to dangerous expansion in use. A microscopic examination is a quick method of determining whether an excessive amount of quartz is present.

An example of difficulties which an examination of the raw materials may solve is shown by the following case where it was proposed to manufacture silica brick from quartz derived from a large vein running through a granite mass. On preliminary tests of three bricks made out of different samples, two bricks failed entirely, fusing to a glassy mass containing the quartz. Subsequent examination of the raw material showed considerable amounts of feldspar and mica present, caused, no doubt, by the material being taken from points too close to the contact of the vein with the granite.

Other properties such as that of the character of the bonding material of brick may also be studied.

Many other ceramic products, such as spark plugs and electrical porcelains, might be mentioned but the foregoing should be sufficient to indicate the many and varied types of investigations to which the petrographic microscope may be profitably applied. Only a beginning has been made and the future should yield much more.

In the foregoing pages an attempt has been made to convey in a simple manner some idea of the relation of petrographic methods to ceramics. That its importance is being recognized by ceramic manufacturers and others is shown by the fact that they are gradually adding to their research staffs men trained

in this field. The field is almost entirely new and capable of important results in the future.

It must not be inferred, however, that these methods are something which may be absorbed in a short time and then put into practice. Articles appear every now and then which show only too well a lack of thorough training and experience. A thorough training in the fundamentals of the behavior of light in passing through crystalline bodies, backed by experience in the application of these principles to the study of rocks and other crystalline substances, is absolutely necessary and is a matter of considerable time if done rightly.

The demand for petrographers for ceramic work presents several difficulties. In the past the supply of petrographers has been ample because their field was confined almost entirely to mineralogical or geological work. Now, however, with the increased industrial demand, the supply is too limited because it represents a narrow special field which is usually taken up only by graduate students. Due to the attention drawn during the war to the vast importance of the mineral resources of the country, there is no doubt but that many more students will in the future be drawn into the allied fields of mineralogy, geology, and mining and that in this manner many more will work their way into petrography than in the past. It is also true that there are only a few universities properly prepared at present to give courses in the latest methods of using the petrographic microscope, and, furthermore, it unfortunately happens that very few of these universities which are able to offer good training have a ceramic school or offer a few courses in ceramics.

One of the greatest difficulties facing the petrographer who goes into ceramic work is the fact that he immediately finds himself in a field about which he has almost no knowledge. The average petrographer comes with a knowledge of mineralogy, geology and perhaps some physical chemistry but with nothing in the nature of ceramic principles and it is a handicap which requires considerable time to overcome. The obvious remedy for this is the institution in the ceramic schools of the country of courses, in connection with the mineralogy departments, which will give every ceramic student at least a fair understanding of the principles of petrog-

raphy and their application to and importance in ceramics. In this way many no doubt will be led to take up graduate work along the lines of ceramic microscopy.

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COLLOIDAL TRI-CALCIUM ALUMINATE.[1]

By A. J. Phillips.

I. Introduction.

In a previous publication[2] it was stated that tri-calcium aluminate, on mixing with water, hydrated to form an amorphous product which later became crystalline. As the aluminate, together with tri-calcium silicate, is responsible for the changes in Portland cement known as initial and final set, a further study of the amorphous aluminate was made with respect to its behavior with large and small amounts of water and to the alteration in its condition caused by the addition of several salts; in order to obtain some idea as to the way in which the set of a normal cement would be affected under similar conditions.

II. Tri-Calcium Aluminate in True Solution.

1.—**Relation between Various Degrees of Dispersion.**—The material used was that prepared by Bates[3] and consisted almost entirely of $3CaO.Al_2O_3$ with entire lack of crystalline outline; a very small amount of $5CaO.3Al_2O_3$ and traces of $2CaO.SiO_2$ and CaO. Before use it was ignited to remove any water and CO_2 present and sifted through a 200-mesh sieve.

A more or less permanent suspension results in case the aluminate is agitated for some time with a large volume of water and the question arose as to whether we were dealing with a true solution of the aluminate with a mechanical suspension of coarse grains or whether we had present a solid phase which was sufficiently subdivided to be in the colloidal state but which was not sufficiently subdivided to be in the molecular state.

It is generally considered that there is no sharp line of demarcation between coarse suspensions and true solutions, but that there is a gradual progression from coarse to fine suspensions,

[1] By permission of the Director, Bureau of Standards.

[2] Bur. Standards, *Tech. Paper* 43 (1914).

[3] *Ibid.*, 78, 6 (1917).

to molecularly dispersed, to ionically dispersed solutions. Apparently then we are unable to draw any dividing line in the case of the aluminate and water, as a coarse suspension is readily formed by shaking with water and Klein[1] has shown that definite crystals of hydrated aluminate are formed after a period of time, indicating that possibly the aluminate has gone into solution and when sufficiently concentrated has crystallized out.

Admitting that both molecularly and colloidally dispersed particles enter into the reactions of the aluminate, we examined first that part in true solution.

2. **Solubility of the Aluminate.**—Suspensions of the aluminate in no case gave clear filtrates when single or double filters were used, or even in the case of a filtration through an alundum cone. By continued filtration the filters clogged so that a clear liquid was obtained; but by the time this had occurred the filtration took place at a very slow rate. By dialyzing a suspension through a collodion sack, first tested with a solution of Congo Red to insure the absence of .pin holes or leaks, a solution was obtained which reacted faintly for alumina and lime. This was considered sufficient indication that a part of the aluminate goes into true solution.

3. **Hydrolysis.**—Hydrolysis occurs whenever we have the salt of a weak acid or weak base in contact with water. The aluminate being the salt of a weak acid and part of it going into solution, we should expect some hydrolysis; especially since Bates[2] has shown that tri- and di-calcium silicates, both salts of a weak acid and a strong base, split off lime hydrolytically.

In referring further to the literature on cement we find several references to the hydrolysis of the aluminate. Rohland[3] states that the aluminate is hydrolyzed to calcium hydroxide and aluminum hydroxide. If hydrolysis does occur however, we should not expect it to be so complete, but would rather expect the separation of free base ($Ca(OH)_2$) and a residue containing less lime than the original material. This follows from the fact

[1] Bur. Standards, *Tech. Paper* **43**, 15 (1914).

[2] *Ibid.*, **78**, 15 (1917).

[3] *Z. ges. Brauw.*, **29**, 704 (1904).

that if a salt of a strong base and a weak acid is only slightly soluble in water, its solubility will be limited by the solubility of the less basic residue formed by hydrolysis. Again in a case of this kind the increasing concentration of free base in solution will tend to check the hydrolysis; at some point, therefore, we will have an equilibrium established depending on temperature, concentration, etc., between the amount of hydrolyzed and unhydrolyzed salt. Therefore, contrary to Rohland, only in a case such as dialysis in which the free base is constantly being removed could we expect to find the aluminate completely hydrolyzed into free base and free acid. This conclusion of course applies to the silicates and Michaelis[1] states that water never removes all the lime, but a simpler hydrated silicate is formed. Desch[2] states that water hydrolyzes both the silicates and aluminates. Reibling[3] refers to the hydrolysis of the aluminate as one of the factors affecting setting time and Allen and Rodgers,[4] during the preparation of the aluminates of sodium, potassium, calcium and strontium in the wet way, found them to be slightly soluble in water and by it decomposed unless the alkali was kept in excess. As hydrolysis is increased by dilution it follows that it will be decreased as the concentration of aluminate to water is increased.

In a normal cement containing 7 per cent of alumina, the latter will be present entirely as tri-calcium aluminate and therefore 18.5 per cent of the cement will be tri-calcium aluminate. Let us suppose that for a normal consistency paste 23 per cent of water will be required; then for each 100 grams of cement there will be 23 grams of water for 18.5 grams of aluminate. Neglecting the water required to wet the grains of the less rapidly reacting material, there is a little over one part of water for each part of aluminate. From this we should expect that on account of the concentration of the solution there would be little chance of hydrolysis.

4. Ratio of lime to Alumina in Solution.—In order to determine the possibility of hydrolysis some of the aluminate suspensions

[1] *Tonind. Ztg.*, **33**, 1243 (1909).
[2] *Cement*, **12**, 192 (1912).
[3] *Philippine J. Sci.*, (*A*) **6**, 210 (1911).
[4] *Am. Chem. J.*, **24**, 304–18 (1900).

were filtered in a bell jar from which the air had been exhausted, the suspension being dropped from a separatory funnel on to a double filter until the latter had choked sufficiently to give a clear filtrate. The first cloudy portions passing were rejected and the solution passing later was analyzed. In no case did the analyses show the ratio of lime to alumina which is called for by the formula $3CaO.Al_2O_3$ (that is 1.647–1.0), but in every case the lime was lower. There was, however, the possibility of the adsorption of the lime by the filter paper which would tend to reduce the lime ratio.

Of more importance, however, was the fact that the ratios obtained were not constant but varied with time. To test the matter further a cylinder of aluminate and water was molded about a brass tube and dried immediately in a carbon-dioxide-free atmosphere. This cylinder, about $1/4''$ thick, $1''$ diameter and $3''$ high, was placed in a soxhlet extractor; water in a flask below was converted into steam and a condenser above permitted the condensed steam to flow into the extractor until it overflowed. The overflow was not returned to the flask and 100 cc. portions were analyzed at intervals. By running the water in and out slowly, the solution obtained was clear and did not require filtration. The results are tabulated in Table 1.

As may be seen, this experiment furnished no evidence of hydrolysis, for if there was, the free base would be soluble in water and consequently the ratios in solution would show an excess of lime instead of a deficiency.

It is known that the purest water, even conductivity water, contains carbon dioxide so that there is the possibility that a layer of calcium carbonate was formed on the surface of the cylinder, but that the penetration of the carbon dioxide did not follow that of the water. Reibling[1] found that when cement in bins, stored in a stock house, was exposed to moist air containing carbon dioxide, the absorption of the latter was limited to a distance very close to the surface while the water was able to penetrate much deeper. However, this explanation assumes the presence of much more carbon dioxide than we have reason to believe was present. The only other explanation is that furnished

[1] *Philippine J. Sci.*, (A) **5**, 397–8 (1910).

TABLE I.

No.	Al_2O_3, grams.	CaO, grams.	Time.	CaO/Al_2O_3 in soln.
1.....	0.0275	0.0302	3 hrs.	1.096
2.....	0.0489	0.0553	4 hrs.	1.131
3.....	0.0670	0.0782	4 hrs. 23′	1.167
4.....	0.0286	0.1016	5 hrs.	1.230
5.....	0.1221	0.1499	22 hrs. 20′	1.226
6.....	0.1375	0.1682	23 hrs. 20′	1.222
7.....	0.1486	0.1826	24 hrs. 20′	1.230
8.....	0.1563	0.1938	25 hrs. 35′	1.240
9.....	0.2450	0.3255	42 hrs.	1.288
10.....	0.2553	0.3391	47 hrs.	1.328
11.....	0.2620	0.3570	52 hrs. 30′	1.354
12.....	0.3168	0.4356	66 hrs.	1.376
13.....	0.3236	0.4473	69 hrs. 25′	1.382
14.....	0.3293	0.4589	71 hrs. 15′	1.393
15.....	0.3366	0.4730	73 hrs. 40′	1.405
16.....	0.3446	0.4870	96 hrs.	1.415
17.....	0.3453	0.5035	119 hrs. 30′	1.449
18.....	0.3570	0.5196	142 hrs.	1.456
19.....	0.3764	0.5519	166 hrs. 30′	1.465
20.....	0.4068	0.6081	9 days	1.496
21.....	0.4189	0.6260	11 days	1.494
22.....	0.4416	0.6620	14 days	1.500
23.....	0.4588	0.6896	18 days	1.503
24.....	0.4718	0.7096	22 days	1.504
25.....	0.4802	0.7292	28 days	1.519

by absorption; a small amount of calcium ions was absorbed from the solution by the solid aluminate, leaving a solution containing less lime than that required by the formula $3CaO.Al_2O_3$. Thus the presence of an excess of free base in solution is prevented by the aluminate and to this extent hydrolysis is possibly increased by absorption. With regard to the amount of aluminate in true solution, a number of tests were made by shaking aluminate with water in such proportions that a large excess of the solid phase was present and the suspension was then dialyzed. The results obtained in this way were somewhat higher than those in which the suspension was filtered through the filter paper but in no case was there obtained a concentration of more than 0.6 gram per liter

at room temperature. We may therefore conclude that the material in true solution plays a very small part in the hydration of the aluminate.

III. Tri-Calcium Aluminate in Colloidal Suspension.

By shaking the aluminate with water, a small amount, as we have seen, is dispersed through the colloidal state and enters into true solution, the major portion, however, remains as a colloidal solution or suspension.

1. **Dispersion of the Aluminate.**—Bates[1] notes that on mixing the aluminate with water, the grains, whether coarse or fine, agglomerate and hydrate only on the exteriors to a film which is not readily permeable to water; consequently the interiors of the grains will not be hydrated. However, if larger volumes of water are used with continuous agitation, the films may be mechanically removed, permitting access of water to the unhydrated material and resulting in swelling to a volume many times in excess of that of the original volume.

Our suspensions were therefore made by grinding the aluminate with water in an agate mortar to break up the lumps, washing through a small 150-mesh sieve, and diluting to the desired volume in a graduated jar. The jars were then rotated in a shaking machine for periods varying from 5 minutes to 8 hours. These suspensions were then allowed to settle until there was no visible precipitation in 24 hours; the cloudy liquid above the settled material was then drawn off and preserved away from CO_2. One suspension which had been kept for eight months was still cloudy and opalescent, the total concentration of aluminate being about 0.5 gram per liter. We may note in passing that an essential characteristic of the colloidal state is that the substance will exist indefinitely as a suspension of solid (or sometimes liquid) masses of very small size in some liquid medium.[2]

It was noted that if a suspension of aluminate was treated with lime water, or if the suspension was made in lime water, that there was an increase in the amount of aluminate in suspension.

[1] Bur. Standards, *Tech. Paper* **78**, 9 (1917).

[2] Burton, "Physical Properties of Colloidal Solutions," **1916**, p. 9.

There was more swelling and less material settling out in a given period of time.

In a suspension of this character, in which we have present material in true solution as well as that in suspension, the addition of the common Ca ion to the material in true solution would tend to reduce its solubility and cause precipitation. On the other hand, the action of the Ca ions on the suspended material would be explainable on the grounds of adsorption and influence on the electrical charges of the particles, resulting in an increased dispersion. As the effect noted was that of increased dispersion—more material remaining in suspension—it is justifiable to disregard the material in true solution and consider only the colloidal material when we arrive at the effect of added salts to the suspensions. Furthermore, when we consider the action of salts on a normal paste in which the amount of water is greatly reduced as compared with the suspensions, the percentage of soluble material will also be greatly reduced and may be entirely neglected. Therefore, in studying the behavior of the aluminate with either large or small amounts of water with added salts, it is necessary for us to consider only the behavior of the colloidal material.

2. **Method of Dispersion.**—Normally-burned tri-calcium aluminate forms a definite crystal, hexagonal or rectangular in outline, belonging to the isometric system,[1] and we would expect it to follow the behavior of a crystalline salt in going into solution. It does not do this however, as microscopical studies have shown. Zsigmondy[2] makes a distinction between crystals and colloids according to their method of solution. A crystal on dissolving gives its outside layer to the liquid so that at any moment the residual piece has the exact composition of the original crystal. A colloid, though giving up particles to the liquid, also takes up considerable amounts of the medium, a process accompanied by swelling. If the solution process is at any time interrupted, the residual colloid has not the composition of the original material. Crystalline lime behaves in the same manner and this is in part attributed to polymerization of the molecule. It is not a simple molecule but is better represented by the formula (CaO)n. The

[1] Shepherd and Rankin, *J. Ind. Eng. Chem.*, **3**, 225 (1911).

[2] "Chemistry of Colloids," **1917**, pp. 8–9.

crystalline aluminate is also probably highly polymerized and in contact with water acts not as a true crystal but as a colloid, going through a gradual change involving several intermediate steps, until the final result is a hydrated crystalline body.

3. **The Tyndall Effect.**—A suspension of the aluminate was filtered through filter paper until a clear filtrate was obtained, but on placing the solution in a glass cell and passing a beam of light through it, the "Tyndall cone," a characteristic of colloidal solutions, was obtained. A comparison with distilled water in the cell showed only a very faintly defined cone.

4. **Dialysis and Diffusion.**—A suspension of the aluminate, having a total concentration of 0.5 gram per liter, was dialyzed through parchment paper for 24 hours. Coagulation resulted with the formation of a deposit full of cracks resembling those in dried mud. In another case a suspension was made by mechanical agitation in water and was kept in a stable condition for three months, hence it is reasonable to suppose that it would not have suddenly coagulated; but on dialyzing for 24 hours through a collodion membrane it settled out inside the membrane to form cracked plates.

The settling out or coagulation is of course due to the diffusion through the membrane of the Ca ions carrying electrical charges which stabilized the suspension.

The dialyzate in each case reacted slightly for Ca and Al ions but the amount of material was too small for exact analysis.

For the diffusion experiment a sol of aluminate was poured on a 3 per cent solution of gelatine which had been allowed to solidify in a test tube. After 24 hours there was no visible diffusion of the sol into the gelatine. In another case the sol was colored with a little Congo Red solution and again there was no visible diffusion into the gelatine.

5. **Electrical Behavior.**—It is known that a particle suspended in water becomes electrically charged through adsorption of an excess of either positive or negative ions from the solution. The sign of the charge may be determined by dipping the points of two wire electrodes into a drop of the dispersoid on a microscope

slide; or by sending a current through it while it is suspended in a U-tube. For our work an apparatus modeled after the Hardy U-tube, described by Burton,[1] was used, the electrodes being of coiled platinized platinum.

A suspension of the aluminate in water did not migrate at all when distilled water was used around the electrodes, but as soon as the water was made a better conductor by the addition of a trace of sodium chloride, the migration was toward the negative pole. At another time a little of the suspension was filtered and this solution used around the electrodes and in a third case lime water was used. In all cases the direction of migration was toward the negative pole, the particles being positively charged.

There was no visible increase in the rate of migration with the addition of either lime water or solid lime hydrate. This was hardly to be expected since we were dealing with a suspension containing an excess of the solid phase which very slowly settled out. Variations in the concentration of lime water added simply affected the amounts of excess solid phase settling out in a given period of time. That is, increasing the concentration of lime water up to saturation resulted in an increased amount of material being held in suspension without affecting the rate at which it would migrate. For instance:

100 cc. H_2O + 1 g. aluminate, shaken 5 min., settled 8 hrs., gave 4 cc. of sediment, while 100 cc. lime water + 1 g. aluminate, shaken 5 min., settled 8 hrs., gave 1 cc. sediment.

Again, it has been shown that polyvalent ions do not generally increase the amount of the charge on particles of the same sign as themselves.[2]

To recapitulate, the aluminate suspension contains a small amount of material in true solution and a small amount present as a mechanical suspension which separates out on long standing. The major portion, however, has the degree of dispersion required of colloidal dispersions which readily pass through a filter, do not diffuse or dialyze, show the development of the Tyndall cone and are electrically charged.

[1] *Phil. Mag.*, [6] **11**, 436 (1906).

[2] Baudouin, *Compt. rend.*, **138**, 1166 (1904); Gee and Harrison, *Trans. Faraday Soc.*, **6**, 42 (1910).

IV. Suspensoid-Emulsoid Relation.

The absorption of water by the fine particles in suspension leads to a consideration of the nature of the suspension; that is, whether we are dealing with a suspensoid, an emulsoid, or both.

In suspensoids we have a disperse phase which does not combine with the medium to any great extent and if there is combination an emulsoid is formed. Obviously, a disperse phase which takes up a small amount of the medium, or which takes up a large amount through a considerable time interval, will have the properties of both suspensoids and emulsoids. For instance, the metallic hydroxides combine to such an extent with water that they are considered more like emulsoids than suspensoids.

One of the striking properties which the aluminate exhibits is its absorption of water and swelling, by capillary attraction, endosmose and molecular embibing. According to Ostwald,[1] when we say that the disperse phase is composed of exceedingly swollen particles or of particles united to a great number of liquid molecules, we imply that its state is liquid. The emulsoid is then a liquid + liquid system which exhibits more of the properties of the medium than it does of the disperse phase. In addition, emulsoids represent an intermediate state between suspensoids and molecular solutions, not because they have a greater degree of dispersion, but because the liquid + liquid system formed resembles in its physical properties, such as density, surface tension and viscosity, those of the medium more than those of the disperse phase.

An interesting confirmation of the presence of an emulsoid sol is found in the light coagulation of the aluminate suspensions. If allowed to stand in a glass jar near a window, the sides of the containing vessel turned toward the light become speckled with grains of the aluminate which cling there instead of settling to the bottom. There is a time effect connected with this, since it occurs for a short time only after the suspension is made and does not occur at all if the shaking of the suspension is continued for some time before allowing it to settle. These grains are suspensoid particles which have been coagulated by the light rays

[1] "Colloid Chemistry," 1915, p. 51.

and the reason why the phenomenon does not continue is that the material still in suspension is being converted into an emulsoid which is not readily affected by light.

The question as to whether suspensoid or emulsoid is present in greater amount in the suspension is unimportant, since the aluminate + water is an unstable system—the direction of change being an initial combination with water and a final crystallization as a hydrated crystal. The intermediate stages such as suspensoid and emulsoid represent varying combinations with water which themselves are unstable.

The aluminate suspension is not coagulated by heat or by alcohol but is coagulated by freezing. It does not form a gel on evaporation or by dehydration over sulphuric acid, the product in each case being a loose powder. Its properties in general resemble those of the sols formed from the metallic oxides: iron, aluminium and chromium.

V. Relation between Normal-Consistency Pastes and Excess Water.

When we attempt to compare the behavior of the suspensions with that of the normal consistency pastes, which we encounter in troweling the aluminate with small amounts of water or in troweling cement with the amount of water necessary for a normal paste, we meet with difficulties because of the fact that larger amounts of water and more mechanical agitation are used in the preparation of the former. It becomes necessary then to show what differences exist between the two and how far we may go in making comparisons.

1. **Effect of Excess Water.**—In a normal paste the amount of water used will rarely be more than 25 per cent of the weight of the cement, while in a suspension it may be 100 times or more. The amount of water used for the suspensions is more directly comparable with the amount used in observing the behavior of cement with water on a microscope slide. Muth[1] has shown that the closer the water approximates that used in actual practice, the less the amount of crystallization in the early periods, or con-

[1] *Cement Eng. News*, **21**, 14 (1909).

versely, the larger the amount of water the more the crystallization. Again, Klein[1] has shown that specimens of aluminate on a slide begin to develop crystals within a very short time, while a specimen molded with 40 per cent of water is very slightly hydrated within 24 hours, the material being amorphous with no signs of crystallization.

Crystallization, as shown for instance in the character of precipitated barium sulphate,[2] depends on concentration. That is, in very concentrated and in very dilute solutions the precipitate will be in the colloidal state so that it will pass through a filter. With solutions of intermediate concentration there is a decrease in the degree of dispersion of the precipitate which becomes crystalline and does not pass through a filter. This applies to variations in the amount of water used with the aluminate. If a large volume of water is used, a sol is formed consisting of ultramicrons surrounded by films of water having a free path and motion, while if small amounts of water are used, as in the normal pastes, a gel is formed consisting of ultramicrons packed closely together; the only difference being in the amount of associated water.

In the case of an intermediate concentration of water, such as is used on the microscopic slide, the dispersion of the aluminate is decreased and crystals of hydrated aluminate are formed. This crystallization effect resulting from decreased dispersion, which in turn is dependent on the relative concentrations of disperse phase and dispersion medium, has been demonstrated by Bates[3] in the hydration of dicalcium silicate. Here it was shown that the amount of water must be rigidly restricted in order to cause the crystallization of lime hydrate from the disilicate.

2. **Effect of Mechanical Agitation.**—It has already been noted that wetting the aluminate results in the formation of a semi-permeable film around the aluminate grains. With mechanical agitation this superficial coating will be broken and allow water to come in contact with fresh surfaces. When we consider the mixing of a sloppy concrete in a mixer, there enters the possi-

[1] Bur. Standards, *Tech. Paper* 43, 15–17 (1914).
[2] Ostwald, "Theoretical and Applied Colloid Chem.," 1917, p. 25.
[3] Bur. Standards, *Tech. Paper* 78, 15 (1917).

bility of the formation of both suspensoid and emulsoid along with the gel. There is sufficient agitation of fine material with water to form a suspension and it follows that there would be no such thing as laitance unless some kind of a suspension were formed.

We then may compare the behavior of the suspensions and normal pastes if we bear in mind that in the suspensions there is a tendency to form a sol while in the pastes the major tendency is toward the formation of an amorphous gel. Microscopical investigations of aluminate and cement pastes[1] have shown that up to 24 hours, which period includes both initial and final set, there is no crystallization but only the formation of amorphous material. Therefore, we may consider that the behavior of the colloidal material in the suspensions is exactly the same as the colloidal material in the normal pastes.

VI. Tri-Calcium Aluminate Gel.

1. **Formation.**—A gel may be formed by a solid taking up the medium or by the coagulation of an emulsoid sol. The degree of dispersion is less than that of a liquid solution and higher than that of the solid from which it was derived. Between these limits the dispersion may assume any value.

In the normal pastes we should suppose that the very fine material would be hydrated and dispersed to a sol which would later coagulate to form a gel with elimination of water, while the coarse material would very slowly hydrate and distend to form a gel.

As a result of ultramicroscopic studies, Zsigmondy[2] states that newly-formed gels are formed of a closely compacted mass of ultramicrons, having structures more or less granular or flocculent, and consisting of a system of islands surrounded by lakes.

2. **The Inherent Water.**—From the rapidity with which a liquid will permeate the entire mass of a gel, it is assumed that the spaces between the ultramicrons are capillary. At the surface of the gel the meniscus of the liquid touching a capillary tube is concave upward and surface tension exerts a pull on the liquid causing it to rise in the capillary. A corresponding and opposite pressure

[1] Klein, Bur. Standards, *Tech. Paper*, **43**, 15–17 (1914).

[2] "Chemistry of Colloids," **1917**, pp. 68–70.

is exerted on the capillary walls, tending to squeeze the ultramicrons together.

Because of the curved meniscus the vapor pressure of the capillary water is greater than the vapor pressure of a plane surface of water.[1] Furthermore, the water associated with the colloid is adsorbed water which is densified and compressed about the ultramicrons. The compression of this adsorbed capillary water results in raising its vapor pressure since as the pressure on a liquid is increased its vapor pressure increases.

Thus in the freshly formed gel we have a pressure exerted on the ultramicrons tending to squeeze them together and also capillary water at a higher vapor pressure than that of normal water; two conditions which have an effect on the relegation or syneresis of the gel.

3. **Syneresis.**—Syneresis consists of the separation of the gel into two phases, a concentrated phase rich in colloid and poor in water and a dilute phase rich in water and poor in colloid; if the gel is left undisturbed for a number of hours and protected against evaporation. The liquid which separates is not pure water but a solution of all the constituents of the gel in both colloid and molecular degree of dispersion.[2] It is the reverse of swelling and postulates the formation and maintenance of the gel for several hours as well as a decrease in the degree of dispersion.

The relegation noted so frequently in connection with the testing of Portland cement is an example of the syneresis of a gel and as noted by Bates[3] is a characteristic of the aluminate. It was found possible to reproduce this phenomenon by stirring 5 grams of aluminate with 5 grams of water in a test tube and slowly adding 5 cc. of 1.21 sp. gr. hydrochloric acid while stirring and cooling the test tube in running water. In a short time the thick paste became solid and if the test tube was inclined and left for 3–4 hours, the syneresis water separated out from the solid residue and slowly solidified to a clear gel which qualitatively showed both alumina and lime. In cases where the tube was not inclined the

[1] Bancroft, *J. Phys. Chem.*, **16**, 395 (1912).

[2] Ostwald, "Theoretical and Applied Colloid Chemistry," **1917**, p. 93.

[3] Bur. Standards, *Tech. Paper* **78**, 10 (1917); *Trans. Am. Ceram. Soc.*, **14**, 558–9 (1914).

gel was always found at the top of the solid residue as though it had been squeezed up. The relegation of cement specimens is more noticeable in th Vicat-ring specimens than in the Gilmore-needle pats, which more nearly fulfills the specification that the gel should be protected against evaporation. The phenomenon has no connection with crystallization but is simply the separation of a watery phase due to pressure exerted within the gel.

In the case of the aluminate gel which is protected against evaporation, the free water will not be lost but the capillary water which has a higher vapor pressure tends to distill out to the surface where its vapor pressure will be normal.

Let us assume that a small amount of this capillary water distills out to the surface, then immediately the radius of curvature of each capillary meniscus will be decreased, surface tension will be increased, the pressure on the ultramicrons will increase, the gel will contract and a further quantity of liquor will be squeezed out. This watery phase does not remain long in contact with the colloid-rich phase, since water is continuously being absorbed by the unhydrated material, so that with time the watery phase is reabsorbed and the mass hardens as a whole.[1]

4. **Coagulation.**—Syneresis is merely one step in the coagulation of a gel and a gradual contraction of the gel takes place continuously. It has been stated that a gel consists of a series of islands surrounded by lakes, such a structure being more or less permeable to water; during the contraction, however, the ultramicrons come into contact with each other, forming a film or cell wall enclosing liquid. That is, we may have the formation of a series of lakes surrounded by islands. Such a structure will be less permeable to water and consequently the loss in water by the gel and further coagulation will be considerably retarded.

As the gel continues to dry out and contract, the effect of increased surface tension becomes noticeable through the tendency of the small particles to assume a spherical shape. This formation of radial spherulites or droplets as a preliminary to crystallization is particularly noticeable with the $5CaO.3Al_2O_3$ under the microscope.[2]

[1] Zsigmondy, "Chemistry of Colloids," **1917,** p. 146.

[2] Klein, Bur. Standards, *Tech. Paper* **43,** 12 (1914).

5. **Variations in Degree of Dispersion of the Gel.**—In concluding this discussion of the formation and coagulation of the gel, we must emphasize the necessity of confining the dispersion within certain limits, in order to control the various physical effects resulting in maximum strength. The hydration to a gel involves the absorption of water, the combination resulting in swelling with a decrease in degree of dispersion. As the amount of hydration is increased, by dispersing the material and offering a greater surface for absorption of water, the material tends to go into solution and the dispersion is increased. We have a constant shifting of the equilibrium, water in colloid $\rightleftarrows$ colloid in water, because of the fact that in hydration most of the water is going into the colloid, and in solution the reverse occurs. Our efforts are confined to getting all the water possible into the aluminate, but owing to variations in the grain size, we cannot do this without getting some of the aluminate into the water.

With regard to the influence of the amount of dispersion on the strength of the hardened material, Bates[1] states that a high percentage of tri-calcium silicate in a cement is undesirable for the development of early strength because of the fact that this material is easily dispersed and remains in a condition in which it readily takes up water in a moist atmosphere and loses it in a dry atmosphere and consequently hardens slowly. It is readily permeable as compared with the aluminate. On the other hand, the aluminate is dispersed with difficulty and is more readily coagulated with the formation of a material having little strength.

6. **Heat Effects.**—On the first addition of water to the aluminate there is an evolution of heat—due to the formation of an exothermic hydrate. This is due to the fact that water is coming into contact with an anhydride. Salts containing all their water of crystallization dissolve in water with absorption of heat and conversely anhydrides dissolve with an evolution of heat. For instance, Thomsen[2] has shown that BaO, CaO and SrO have positive heats of solution, $Ca(OH)_2$ has a very small positive heat of solution, while $(BaOH)_2.8H_2O$ has a negative heat of solution.

[1] Bur. Standards, *Tech. Paper* 78 (1917).

[2] *Ber.*, 16, 2613 (1883).

The heat evolved by the aluminate will depend upon its previous treatment with regard to exposure to water vapor. That is, the sudden large evolution of heat on mixing the aluminate with water may be lessened or eliminated by causing it to first combine with a small amount of water; by exposing it to moist air, shaking with wet sand, exposure to an atmosphere of steam, etc.[1] In this case, of course, we are dealing not with an anhydride but with a hydrate.

In addition, the solubility of calcium aluminate is greater in cold than in hot water; the absence of a heat effect will therefore favor a greater concentration of aluminate in true solution, thus more rapidly supplying ions to effect dispersion. In aerated cements the absence of a heat effect will likewise favor the solution of calcium sulphate.

It has been shown that in the dissolution of dry colloids there are two heat effects, hydration, an exothermic change, and solution, an endothermic change.[2] Gelatine, gum arabic, gum tragacanth, dextrine, starch, etc., behave like anhydrous salts. Conversely, the aluminate behaves like a dry colloid in evolving heat on hydrating, for the elimination of a heat evolution by preliminary aeration points to the probability that the taking up of water in this case is analogous to the imbibition of water by a dry gel.[3]

In addition to the heat resulting from the formation of a hydrate, there is a disturbance of the equilibrium $(H_2O)_n \rightleftarrows nH_2O$ of the water molecules, which causes an evolution of heat. This is also eliminated when the aluminate is previously aerated because the contact of water with a solid having even the thinnest film of liquid condensed upon it does not give rise to a disturbance of this equilibrium.[4]

If there is sufficient positive-ion concentration to disperse the hydrated aluminate as fast as it is formed, the resultant of the heat effects will be either zero or negative, since the dispersion of the aluminate is accompanied by an absorption of heat. An

[1] Reibling, *Philippine J. Sci.*, (A) **5**, 396 (1910); Candlot, "Ciments et Chaux Hydraulique," p. 349; Bamber, *Concrete* (Concrete-Mill Section), **23**, 23–4 (1916).

[2] Wiedmann and Ludeking, *Ann. Phys. Chem.*, [2] **25**, 145–53 (1885).

[3] Taylor, "Chemistry of Colloids," **1915**, p. 156.

[4] Gaudichon, *Compt. rend.*, **157**, 209 (1913).

explanation of the absorption of heat may be found in a consideration of the Principle of Mobile Equilibrium, a generalization first given by Le Chatelier and Braun.[1] When a factor determining the equilibrium of a system is altered, the system tends to change in such a way as to oppose and partially annul the alteration of the factor. For instance, in the compression of a volume of gas the volume is decreased and according to the principle the system should oppose this volume decrease. It does this by giving rise to an evolution of heat, for the temperature rise tends to make the volume increase. Conversely, if the volume is increased, the temperature will fall.

During the dispersion of the aluminate, involving an increase in volume, or better an increase in surface, the temperature will fall with absorption of heat as this will tend to contract the volume and make it smaller. In the coagulation of the aluminate, which involves a decrease in volume or surface, there is an evolution of heat because the latter tends to oppose the decrease in volume.

VII. Adsorption or Absorption.

The control of hydration is affected to a considerable extent by the adsorption of electrolytic ions. This leads to a consideration of whether we are dealing with adsorption or absorption. When a gas or liquid bathes the surface of a solid, it must either accumulate in a layer on the outer surface or penetrate the latter to a certain degree. In cases where condensation occurs only on the outer surface or in a very thin layer of the surface, the phenomenon is termed adsorption. If there is penetration to the interior it is termed absorption. The essential difference is that in the first case the composition of the material is different on the surface from the rest of the mass while in the second case the composition of the entire mass is changed.

In examining the aluminate we find a good example of adsorption, for the treatment with water changes the composition of the outer surface only, a hydrated film being formed while the interior is unchanged. Any ion activity will therefore be confined to the surface. It is not to be considered that no absorption takes place, for the rupture of the films by mechanical treatment or the swelling

[1] Lewis, "System of Physical Chemistry," **2**, 140–1 (1916).

of the particles will allow the penetration of liquid to the interior. However, under the circumstances with which we are dealing, the first contact of liquid with the solid particles results in the formation of a film and therefore the major effects are surface effects and due to adsorption.

VIII. The Effect of Calcium Hydroxide.

1. **Formation of the Suspension.**—Thus far we have considered the aluminate alone; however, in cements we find the aluminate functioning in the presence of a hydrated-lime solution formed either from the free lime in the cement or from lime split off from the tri-silicate. Therefore we shall consider the effect on the aluminate of a lime-hydrate solution.

It has been previously stated that lime water effects a greater dispersion of the aluminate than does pure water. This is due to the fact that the positive calcium-ions of the lime hydrate are adsorbed by the hydrated aluminate. When the concentration of adsorbed ions becomes sufficiently great, or when a sufficient number of ions have been adsorbed, their charges repulse each other and the aluminate is dispersed. In order to get the repelling action we must suppose that the positive calcium-ions are adsorbed more than the negative hydroxyl-ions. The results show that this occurs or in other words there is a preferential adsorption of one kind of ion.

If we are thus able to demonstrate the formation of a stable sol by adsorption of like charged ions, then in the formation of a sol simply by mechanical dispersion in water something in the nature of a like process must take place. In the case of aluminate and water alone, a small amount is dissolved to form a true solution and from this solution the hydrated aluminate adsorbs ions and is dispersed.

Since water adsorbed by the aluminate grains is densified and compressed by reason of the adsorption, any salts in solution will likewise be compressed and their solubility increased. If the positive calcium-ions are adsorbed, the negative hydroxyl-ions are left in higher concentration around the grains than in the rest of the solution. They will therefore tend to diffuse away into the surrounding liquid in order to equalize the concentration.

2. **Coagulation of a Suspension.**—The aluminate has a limited capacity for the adsorption of positive ions, so if the concentration of negative ions is sufficiently increased, at some concentration of the latter, the positive charges on the aluminate will be neutralized by the adsorption of the negative ions because of their increased concentration. This will result in the precipitation of the uncharged aluminate, however, as the charge is neutralized and the material coagulates; the adsorbing surface is decreased and consequently the ions which were held by adsorption are given back to the solution to exert a dispersive effect on undispersed material. For this reason the dispersive effect will constantly be working against the coagulating effect, which explains why the concentration of negative ions must be so increased. The process being a reversible one, we may express it by the following reactions:

$$\overline{CaAl} + Ca(OH)_2 = \overline{CaAl}\,Ca(OH)_2$$

$$\overline{CaAl}\,Ca(OH)_2 \rightleftarrows \overline{CaAl}\,Ca + 2\overline{OH}, \quad \text{where } \overline{CaAl}$$

represents an ultramicron of calcium aluminate without regard to size, form or composition.

In order to obtain a precipitating effect with lime hydrate, the concentration must be very greatly increased because of its slight solubility in water. A much weaker solution of sodium hydroxide may be used to show the precipitating effect largely because in this last case we have to overcome the stabilizing effect of a monovalent cation while in the former case we are dealing with a divalent cation.

3. **Effect on the Normal Pastes.**—It is of interest to note the behavior of hydrated lime with the aluminate and the restricted amount of water required for normal-consistency pastes. We should expect it to exert a dispersing action in small amounts; however, it had no effect in preventing a flash set from taking place, but on working the paste for several minutes to overcome the granulation, a considerable retardation in the time of set was noted. This is, of course, similar to the flash set observed when unplastered cements are treated with water.

A pat of aluminate with 80 per cent of water became hard enough to support the Gilmore needle in four hours, while by

substituting lime water for distilled water, initial set was not attained in forty-eight hours. By increasing the percentage of hydrate very considerably the setting time was diminished as follows:

5 g. aluminate —4.0 cc. H_2O	Set in 4 hrs.
5 g. aluminate —0.05 g. $Ca(OH)_2$ —4 cc. H_2O	Set in 27 hrs.
5 g. aluminate —4.0 g. $Ca(OH)_2$ —7 cc. H_2O	Set in 1 hr. —40 min.

This illustrates the effect of increasing the total ion concentration over and above the limit of the adsorptive capacity of the aluminate for positive ions.

The tests, while not comparable with the larger amounts of cement and water used for normal pastes as regards period of working, radiation of heat, etc., are comparable among themselves and suffice to show a difference between the presence of large and small amounts of hydrated lime.

Spackman[1] has noted that the effect of mixing 90 per cent of hydrated lime with 10 per cent of aluminate is to increase the rapidity of hardening. He states in addition that it causes the lime to become colloidal or form solutions in which the lime is suspended. This is, of course, an indication that the aluminate adsorbs the lime and is dispersed. It may also be noted that neither hydrated lime nor burned lime form permanent suspensions in water. In fact, they settled so rapidly that we were unable to determine whether the material migrated under the influence of the electric current.

We find references all through cement literature to the effect of lime in causing hydraulic qualities to be developed. For instance, Michaelis,[2] in considering the hardening of cement, states that when a sufficient concentration of lime hydrate is reached, a gel is formed. This, as we have explained, is the concentration of positive ions necessary to cause dispersion. Again Zulkowsky[3] states that barium, strontium, calcium, sodium and potassium oxides bring out the hydraulic properties of blast furnace slag. The significant statement is made that "very basic slags need no alkali addition," due to the fact that a part of the material goes

[1] *Proc. Am. Soc. Testing Materials*, **10**, 318 (1910).

[2] *Tonind. Ztg.*, **33**, 1243 (1909).

[3] *Ibid.*, **22**, 285, 316, 363 (1898).

into true solution, ionizes, and furnishes sufficient concentration of ions to disperse the material.

Further, the statement is made that these alkali additions do not combine, for they may be washed out from the hardened cement. This is an indication, as has already been pointed out, that ions are adsorbed by the immense surface furnished during dispersion, but as soon as the surface is decreased by coagulation, the adsorbed ions are released and may be readily removed.

Finally, there are numerous patented processes involving the treatment of molten slag with solutions of the alkali and alkaline earth salts, as well as salts of iron, aluminium and chromium, for the purpose of securing a hydraulic material. The multiplicity of salts used can only be explained through the adsorption of ions of the same kind of electrical charge, their mutual repulsion causing dispersion and the further adsorption of oppositely charged ions to neutralize the previously adsorbed ions thus causing coagulation.

IX. The Effect of Calcium Sulphate.

1. **Effect on the Suspension.**—In shaking the aluminate with water and varying percentages of plaster, from one to twenty per cent, a very slight increase in the rate of migration was noted. It was almost inappreciable and experiments along this line were discontinued as in all cases the suspension remained positively charged.

With increasing amounts of plaster, other conditions remaining constant, there was a greater amount of sediment settling out in a given period of time. With no plaster, a fine-grained sediment was secured which was difficult to filter and which clung tenaceously to the filter paper. As the amount of plaster was increased, the precipitated material became more flocculent, the size of the flakes settling out became larger, and filtration became easier. This was especially noticeable with 10–15 per cent of plaster. With smaller amounts the material settled out, leaving a cloudy, opalescent liquid above but with over 10 per cent the supernatant liquid clear and transparent. The character of the sediment also changed, as with small amounts of plaster the precipitate settled out in a hard mass which was removed only by scraping the bottom of the glass jar with a rod, while as the plaster was in-

creased the sediment became softer and could be poured out along with the liquid.

2. **Adsorption of the Sulphate Ion.**—At no time did the filtrates from the suspensions give a reaction for sulphate even in cases where as much as 20 per cent of plaster was used. This phenomenon in connection with cements has been noted by a number of investigators. Bates[1] notes that the plaster in cements is held in a form which prevents its removal by distilled water and further in the treatment of cements or aluminate with sulphate solutions that very much more is fixed or adsorbed than is necessary for the formation of tri-calcium sulpho-aluminate. Candlot[2] states that if cements are shaken with water and the latter drawn off and analyzed, there is a decrease in the amount of sulphate present up to the time of set, when there is none present. Also Deval[3] notes that hydrated cements seem to retain sulphate which cannot be washed out with water.

3. **Retrograde Adsorption.**—If an adsorbing surface change through agglomeration, crystallization, etc., its adsorptive power will change and the adsorbed salt will be less firmly held.[4] This action, which is called retrograde adsorption, has been demonstrated by Hase[5] in regard to the adsorption of methylene blue by colloidal sulphur, the blue being returned to the solution as the sulphur changes its degree of dispersion. Again Wagner[6] has shown that in the hydrolysis of the salts of aluminium, iron, etc., the liberated acid is first adsorbed by the dispersed material, but as the particles increase in size, the adsorption is diminished and the liberated acid is returned to the solution.

We were concerned with this retrograde adsorption on account of the gypsum crystals noted in hardened cements. Apparently either the sulphate has been incompletely combined with the aluminate during mixing and crystallizes at some later time, or else when the aluminate crystallizes with decrease in its degree

[1] Bur. Standards, *Tech. Paper* **12**, 36–7 (1912).
[2] "Ciments et Chaux Hydraulique," p. 323.
[3] *Bull. soc. encour. ind. nat.*, **101**, 96 (1901).
[4] Bancroft, *J. Phys. Chem.*, **18**, 549 (1914).
[5] *Kolloid-Z.*, **17**, 153 (1913).
[6] *Monatsh.*, **34**, 95 (1931).

of dispersion the adsorbed sulphate is given up and eventually crystallizes.

To test the matter, a number of samples of aluminate with 10 per cent of plaster and a large excess of water were shaken for one day, allowed to settle for varying periods of time, and the filtrates from these suspensions analyzed for sulphate. The periods selected were 1, 3, 7, 14 and 28 days and in no case was a positive reaction for the sulphate ion secured although the aluminate had visibly agglomerated. Thinking that possibly the amount of water used had not been favorable to the crystallization of the aluminate, a sample which had been treated as above and the suspension allowed to stand for four months, was slowly evaporated over sulphuric acid in a vacuum desiccator. The evaporation to apparent dryness required one week's time and the water solution was examined at intervals for the presence of sulphate. As none appeared in solution, the moist residue was shaken with water and a small amount filtered off. As no sulphate was found here, the residual material was dialyzed and in the dialyzate a satisfactory reaction for sulphate was secured.

As we shall later show, the aluminate is able to adsorb undissociated salts from solution. The adsorption of undissociated calcium sulphate, which may hydrate in place and later crystallize, eliminates the possibility of determining whether or not the sulphate has been completely or incompletely incorporated with the aluminate during mixing.

4. **Coagulation.**—It is known that increasing amounts of plaster or gypsum affect the coagulation or setting time of cements in various ways. For instance, Reibling[1] states that as the percentage of plaster is increased, the set takes place earlier, later, and then earlier. No adequate explanation of the cause of these changes has ever been given but we shall attempt to show that they may be readily explained by a consideration of the colloidal state of the aluminate.

For testing the rate of coagulation, pats were made consisting of aluminate, water, gypsum or plaster and hydrated lime. The water at 20° C in all cases was equal to 50 per cent of the weight

[1] *Philippine J. Sci.*, (*A*) 6, 226 (1911).

of aluminate and the hydrated lime equal to 2 per cent of the same. The mixing period was limited to exactly one minute and the percentage of sulphate added was plotted against the setting time as shown in Fig. 1.

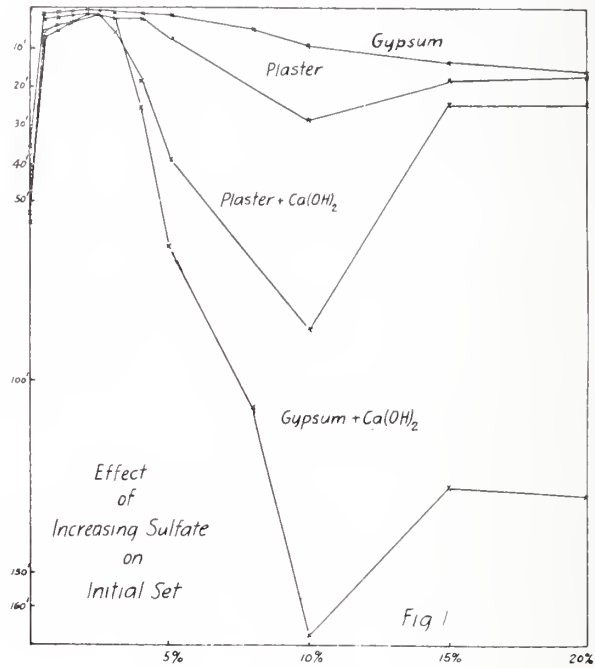


FIG. 1.

4*a*. **The First Acceleration.**—If the aluminate is mixed with water an exothermic hydrate is formed, heat is evolved, and coagulation follows the evolution of heat. However, if the pat is troweled following the flash set, the coagulated hydrated film is broken, positive ions are adsorbed from the aluminate in solution, and the hydrated aluminate is dispersed. The setting time or coagulation then takes place at a later period. In the case of the pats to which no hydrated lime was added, the initial set was retarded to 38 minutes as compared to 53 minutes with those pats in which 2 per cent of hydrated lime was present.

With the addition of plaster in small amounts, the change in

viscosity which allows the pat to support the needle indicating initial set, takes place sooner. The time of set is accelerated to a minimum so that coagulation and evolution of heat takes place while the material is still being mixed. There is this difference, however, from the pats containing no sulphate, in that the latter, while setting at once were reworked and the set delayed, but in the case of those containing up to 3 per cent of sulphate, they remained a crumbly, loose powder even with continued working and could not be formed into a pat.

The setting time in every case is a resultant from the forces tending to disperse the material and those working toward coagulation. Up to 3 per cent plaster or gypsum, with or without hydrated lime, the coagulating forces are in the ascendency and are so powerful as to prevent dispersion of the material by reworking to break up the coagulated film.

Dehydration Effects.—With amounts of plaster or gypsum up to 3 per cent, the set took place in 1–2 minutes. As there was not sufficient elapsed time for the sulphate to go into solution and crystallize out as the di-hydrate, we may eliminate this effect at once. There remains then the probability of a dehydration; a part of the water associated with the aluminate was requisitioned by the sulphate which is able to combine it to form a chemical hydrate—the amount of water necessary to take all of the sulphate into solution being in excess of the total water furnished for both aluminate and sulphate.

We assume that there is a dehydration of the aluminate for the following reasons: Though ions and molecules are admitted to hydrate in solution, it is not conceded that the water in less dispersed material is stoichiometrically combined. In this latter case it is considered that the water molecules are simply gathered more closely around and compressed about the particles.[1] The fact that the water in gels may be replaced by carbon disulphide, ether, alcohol, etc., without any great change in the character of the gel, points to the fact that the water is not chemically combined but is present as adsorbed water filling the spaces between the ultramicrons.[2]

[1] Tyrer, *J. Chem. Soc.*, **99**, 871 (1911).

[2] Zsigmondy, "Chemistry of Colloids," **1917**, p. 139–40.

The water in the colloidal aluminate formed in the early stages of hydration is therefore not chemically combined but merely associated water; there is little or no affinity between the two while the water in the gypsum is chemically combined, indicating considerable affinity. According to Cavizzi,[1] calcium sulphate in solution forms a stable hydrate containing much more than 2 H_2O.

Although there is not sufficient water present to take all of the sulphate into solution and form any such hydrate, the affinity for water exists and the sulphate will strive to secure the necessary water. The partition of water between the aluminate and sulphate will be but slightly affected by the relative proportions of the two, but will be largely influenced by the chemical affinity of the latter for water, an affinity which is lacking in the colloidal aluminate.

The dehydration of a heavily hydrated material involves at first an increase in the degree of dispersion, the swollen particles become smaller due to removal of associated water. If dehydration is continued, as in the case of an evaporation to dryness, the increased dispersion is followed by a decreased dispersion. There is an agglomeration of the particles with decrease in the total surface. The aluminate is not heavily hydrated in two minutes' time, so that the chief effect of the plaster will be a dehydration resulting in agglomeration of the particles with a decrease in dispersion. This is what occurs in "flash set." The effect is not due to the fact that plaster is partially dehydrated as compared with gypsum, for the latter in amounts up to 4 per cent has the same effect.

Rohland[2] has called attention to the acceleration of the initial set of cements by the addition of small amounts of calcium and aluminium sulphates. It will be noted that both of these salts crystallize with water of crystallization and it has been found that the larger the amount of solvent combined as solvent of crystallization, the larger is the amount held in combination as a hydrate when in solution.[3] This of course postulates a desire or affinity

[1] *Gazz. chim. ital.*, [1] **44**, 448 (1903).

[2] *Kolloid-Z.*, **8**, 251 (1906).

[3] Jones and Getman, *Am. Chem. J.*, **31**, 303 (1904).

on the part of the salt for water even though it is not in solution.

4*b*. **The First Retardation.**—In the addition of the above salts in small quantities the chief effect noted is that resulting from the desire to associate a large number of water molecules with themselves. When we add more than 4 per cent of plaster or gypsum under the conditions noted, we begin to see the effect of the ion dispersion already noted under the treatment of the aluminate with hydrated lime; in that the plasticity of the paste is increased and the heat evolved is less. The presence and adsorption of an increased number of positive ions gradually overcomes the coagulative effect caused by dehydration, the resultant being a retardation of the speed of coagulation.

The introduction of 2 per cent of hydrated lime along with the plaster or gypsum has a marked effect in still further retarding initial set. This is because we have added a divalent cation with a monovalent anion and the concentration of the latter must be considerably increased before its negative charge will neutralize the first adsorbed positive ions. It is obvious that 5 per cent of hydrate with 5 per cent of plaster will be more effective in retarding the set than will 10 per cent of plaster, although the same number of positive calcium ions will be present in each case. The curves for the changes in setting time with increasing concentrations of plaster and gypsum are shown in Fig. 1.

Rate of Addition and Solution of Retarder.—Two factors enter into the dispersion of the aluminate by the addition of positive ions—the concentration of the ions and the rate at which this concentration is attained. It has been found that the coagulation of a sol depends not only on the concentration of the added salt but also upon the rate at which it is added.[1] For instance, arsenic sulphide sols are much more indifferent to a slow addition of barium chloride than to a sudden addition. The same condition applies to the dispersion of the aluminate, in that the latter will absorb water and flocculate, but if there is an electrolyte dissolved in the water, it will adsorb a more or less concentrated solution of electrolyte and thus reduce the proportion of adsorbed water. The same result is attained if the aluminate is agitated

[1] Burton, "Phys. Properties of Colloidal Solutions," 1916, pp. 148–9.

with a sufficient amount of water for some time, in that some of the aluminate goes into true solution and thus furnishes an electrolyte in contact with the undissolved material. Furthermore, if the positive-ion concentration in solution is high enough a dispersive (deflocculating) effect is secured in opposition to the coagulation which is secured by the use of pure water. Thus the rate at which the positive ions go into solution as in the case of a difficultly soluble salt and the rate at which the necessary concentration of these ions are brought up to the aluminate are the two chief factors in causing a retardation of the set.

In comparing plaster and gypsum, the former dissolves faster and it would seem reasonable to suppose that 5 per cent of plaster would be more efficient than 5 per cent of gypsum in effecting dispersion. This was found to be true, the plaster retarding the set to 9 minutes while the material containing gypsum set in 2 minutes. It must be borne in mind, however, that we did not allow for the water in the gypsum but simply added 5 per cent of the weight of the aluminate in either case. A comparison of the actual SO_3 content of each would tend to bring the results closer together.

4*c*. **The Second Acceleration.**—As the concentration of plaster is increased beyond 10 per cent, the neutralizing action of the negative ions is exerted and the setting time tends to be accelerated. In addition an acceleration caused by the setting of the plaster itself becomes distinctly noticeable.[1] This acceleration does not occur to such an extent with gypsum and the rise in the curve with hydrate is partially due to the fact that the water added was kept constant in all the specimens and as the amount of retarder was increased the ratio of water decreased.

4*d*. **The Second Retardation.** *Salt Peptization.*—As the amounts of plaster or gypsum are increased over 15 per cent the initial set is again retarded and the final set, which had previously immediately followed initial set, is also delayed, the interval in some cases being as high as fifty minutes. That we have a retardation is evidenced by the shape of the curve, which should

[1] Meade, "Portland Cement," 1903, p. 309; Reibling, *Philippine J. Sci.*, (A) 6, 229 (1911).

follow a straight line if we consider that as increasing amounts of plaster are added the set should be correspondingly decreased, the setting time tending to approach that of plaster alone.

This retardation is due to the adsorption of un-ionized salt. We are dealing with an un-ionized salt in these cases because the concentration is too great to allow of any considerable dissociation taking place. We encounter then the effect resulting from the adsorption of both ions of the salt which results in a kind of dispersion known as salt peptization.

We start with an ion peptization because one ion is adsorbed more than the other; with increasing salt concentration the adsorption of the first ion (Ca) varies but slightly with the concentration while the adsorption of the second ion (SO_4) continues to increase relative to the first, until we get neutralization of the first and coagulation. At the same time the adsorbed salt is tending to peptize the material so that with still greater salt concentration we should expect salt peptization to overcome coagulation. The possibility of peptization by an adsorbed salt is generally overlooked because an increase in concentration of peptizing salt is apt to cause coagulation.[1]

The slowing up of both initial and final set thus results from the adsorption of increasing amounts of sulphate, the peptizing effect resulting from this adsorption acting in opposition to the coagulating effect. The effect of this adsorption is especially noticeable in the suspensions of aluminate in water. With increasing amounts of plaster, coagulation was effected in every case but a soft, mushy precipitate was obtained, constantly increasing in volume as the sulphate was increased. Bates[2] calls attention to the fact that the addition of increasing amounts of sulphate to cements has a tendency to make the initial set quicker and the final set slower. This salt peptization is not confined to calcium sulphate, as it has been shown that a number of compounds such as tannin, straw infusion, oxalic acid, quinoline, etc. all exert a retarding effect on the final set of cements.[3] The effect here is not due to a high concentration preventing dissociation

[1] Bancroft, *J. Phys. Chem.*, **20**, 102–3 (1916).

[2] *Proc. Am. Soc. Testing Materials*, [11] **15**, 126 (1915).

[3] Benson, Newhall and Tremper, *J. Ind. Eng. Chem.*, **6**, 795 (1914).

but to the fact that at any concentration these salts are but very slightly dissociated.

Heat Effects.—With the normal pastes in which plaster or gypsum was used without any lime hydrate, at the lower concentrations there was a noticeable evolution of heat before the material set. With 2–4 per cent of plaster a smooth paste could not be obtained, but when 2 per cent of hydrate was added, a plastic paste was obtained with all concentrations of sulphate—the heat developing just before or at the time of set. It should be noted that 2 per cent of hydrate is sometimes added to cements containing 18–20 per cent of aluminate; the addition of 2 per cent of hydrate to 100 per cent of aluminate in our case represents a much smaller ratio and at the same time it was very effective. In the same way an addition of 20 per cent of plaster to 100 per cent of aluminate represents an addition of about only 4 per cent to a normal cement.

With amounts of plaster and gypsum exceeding 10 per cent the heat evolution was distinctly noticeable about three minutes after initial set had taken place, showing that the heat evolved was the result and not the cause of coagulation of the gel. This, as has been shown, is due to an effort on the part of the system to resist a contraction in volume.

In view of the erratic heat effects noted above in connection with the coagulation of the gel it seems unlikely that there is any connection between a maximum evolution of heat and the time of initial set. Various experimenters have determined the heat rise for cements but with one exception the presence of interfering elements has prevented their curves from assuming any one general shape.[1] In a normally-setting cement there may be a rise due to hydration which is compensated by an adsorption of heat accompanying dispersion. In cases where the final set is delayed, the heat of coagulation is slowly disseminated and no great rise is noted, whereas, in the case of a quick coagulation the positive heat effect will be much greater than any of the effects hitherto

[1] Taylor, "Practical Cement Testing," p. 92; Gary, *Concrete Constr. Eng.*, **1906**, p. 1,350; Hossbach, *Tonind Ztg.*, **33**, 1083 (1909); Watertown Arsenal Reports, **1901**, p. 493; Cushman, *J. Ind. Eng. Chem.*, **4**, 728–30 (1912).

noted. In the case of a continuous agitation of the cement paste, as proposed by Beals,[1] who secured curves of the same general shape, the continued agitation of the cement will cause an increase in the adsorption of heat due to increased and continued dispersion. The rise in the curve indicating coagulation will therefore not correspond with the initial set of a normal paste because of the continued dispersion which is effected.

The Fineness of the Plaster.—The plaster used to secure the curves shown in Fig. 1 was that portion passing a 300-mesh sieve and, in cases where gypsum was used, it was ground under alcohol, passed wet through a sieve, dried at 50° C, and sifted through a 300-mesh sieve by tapping. A microscopical examination of the powder showed it to have the same refractive index as gypsum.

Rohland[2] notes, concerning the addition of retarders, that in general with equal additions, dissolved substances will have a stronger effect than solid difficultly-soluble materials. So that the finer the retarder is ground, in the case of plaster, the nearer will it approach the properties of a liquid. In adding calcium sulphate to cement in order to retard the set, it is customary to add it while crushing the clinker in the ball mills; its progress, then, through the clinker tube mills (or their equivalent) will result in its being ground very fine as it is softer than the clinker. To make up for this dispersion of the sulphate during grinding, we pulverized the material separately.

The results shown in Fig. 1 are open to criticism on account of the fact that the water was not varied as the percentage of solid material was increased—which would cause set to take place sooner than otherwise. In order to show that increased adsorption follows as the retarding salt is increased in fineness, a large sample of plaster was sifted through a nest of sieves and the material remaining on each sieve was used with aluminate in each case. A constant quantity of water, plaster and aluminate was used; 10 per cent of plaster, 50 per cent of water and 2 per cent of hydrate in cases where the latter was employed. The only variable was the grain size of the plaster.

The results are shown in Fig. 2 in which the setting time in

[1] *Concrete-Cement Age*, **3**, 45–6, 52–9 (1914).

[2] "Der Portland Zement," **1903**, p. 13.

minutes is plotted against the fineness of plaster. The effect of the addition of hydrated lime is particularly noticeable with the plaster retained on the 240-mesh sieve. Without hydrate the set is delayed to a maximum and with finer plaster its own set begins to affect the setting time of the material. With the addition of 2 per cent of hydrate, the dispersion of the aluminate is much increased and this dispersive action prevails to a greater extent over the coagulative forces.

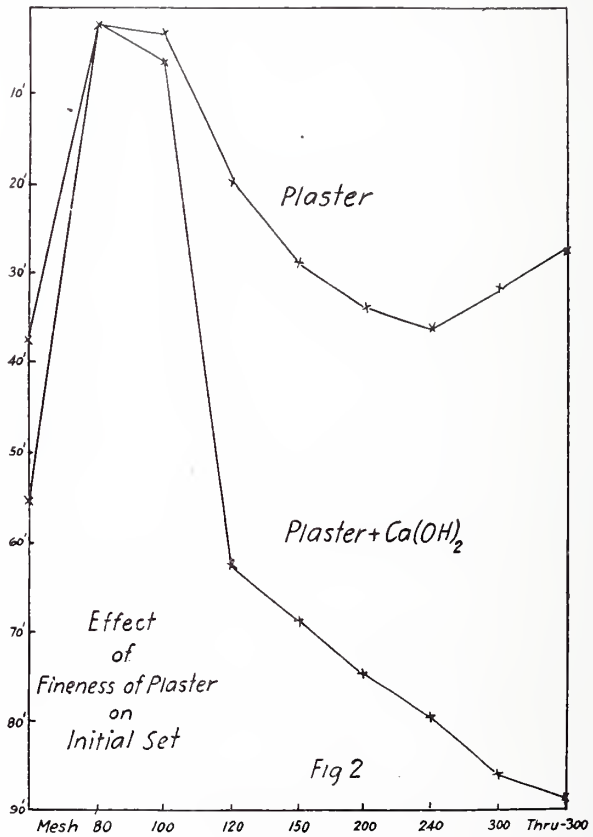


FIG. 2.

The Character of the Pats.—The pats resulting from the tests of the aluminate, as shown in Fig. 1, were in some instances

placed in cold water immediately after initial set and in other cases were kept 24 hours before immersion. As they were not visibly affected after one week's immersion, they were given the customary five hours' bath in steam and in no case did they become soft and mushy. With plaster or gypsum under 10 per cent there was a development of fine cracks which was especially marked in the samples containing only 2–3 per cent of retarder, but those having over 10 per cent were sound and hard. In some cases the surface was chalky but underneath this surface-layer the pat was hard and not readily scratched.

Those pats remaining from the tests, shown in Fig. 2, were sound in cold water but after a time developed radial cracks which were especially noticeable in the coarse plaster samples. A microscopic examination of the hardened pats showed that in every case gypsum crystals were present. In those containing the coarse plaster, the aluminate grains showed dark centers of unhydrated material, a ring of hydrated material, and the gypsum crystals arranged around the outside of this ring. As the plaster increased in fineness, the unhydrated material grew less and the gypsum crystals were scattered through the gel instead of being ranged around the outside. The increased fineness of the plaster therefore resulted in increased adsorption by and better dispersion of the aluminate.

X. Summary.

1. Tri-calcium aluminate, with a large excess of water, forms a suspension containing small amounts of coarsely dispersed and molecularly dispersed material, the major portion being dispersed to a degree comparable to those colloidal suspensions which readily pass through a filter, do not diffuse or dialyze, show the development of the Tyndall cone and are positively charged.

2. The colloidal aluminate is not coagulated by heat, is coagulated by freezing, has a large capacity for the adsorption of ions and undissociated salts and its properties in general resemble those of the sols formed from the metallic oxides Fe_2O_3, Al_2O_3 and Cr_2O_3.

3. With the restricted amounts of water used in cement pastes, the aluminate is converted into and remains as a gel, at least during the first 24 hours after mixing.

4. The rate of dispersion of tri-calcium aluminate to the gel state is influenced by its previous exposure to moisture, the amount of heat developed during hydration, the adsorption of positive ions, the valency of the ions and the amount of dissociation of the salts furnishing these ions.

5. The rate of coagulation is affected by the amount of dispersion effected, the syneresis of the gel, the neutralization of previously-adsorbed positive ions by negative ions, the adsorption of undissociated salt and with the addition of plaster, by the fineness and set of the plaster itself.

XI. Acknowledgments.

It is a pleasure to state that this problem was suggested by and the work carried out under the direction of Mr. P. H. Bates of the U. S. Bureau of Standards, to whom acknowledgment is hereby made. We are also indebted to Mr. A. B. Peck for the examination microscopically of a number of specimens and to Mr. J. C. Evans for assistance in the analytical work.

PHILADELPHIA, PA.

THE INFLUENCE OF TEMPERATURE ON THE TRANSMISSION-FACTOR OF COLORED GLASSES.

By M. Luckiesh.

There are numerous gaps in our knowledge of the physics and chemistry of colored glasses which either have not been spanned at all or have been flimsily bridged by theory supported by inadequate experimental data. It appears to the writer that physical investigations of many of the phenomena pertaining to colored glasses are likely to be most fruitful sources of information, especially if these are carried out in parallel with chemical investigations. It is not the purpose of this brief note to discuss the field in general but to present one of the many interesting phenomena pertaining to colored glass which doubtless will be explained eventually through physical investigations.[1] Hyde, Cady, and Forsythe,[2] in studying red pyrometer-glasses, noted the influence of temperature on the transmission characteristic of a red glass and investigated this influence for temperatures from 20° to 80° C. The transmission-factor of the red glass was found to be appreciably less for various wave-lengths at the higher temperatures than at the lower temperatures.

It appeared of interest to ascertain how generally the transmission-factors of colored glasses were affected by temperature. In a preliminary study it did not appear worth while to investigate this question spectrophotometrically,[3] therefore only the transmission-factor for total visible radiation was considered. However, an idea of the change in spectral transmission is gained through the change in color of the specimen as its temperature is altered. It is hoped that at a later date a careful study of this

[1] M. Luckiesh, "The Physical Basis of Color Technology," *J. Frank. Inst.*, **184**, 227 (1917).

[2] Hyde, Cady and Forsythe, "Effective Wave-Length of Transmission of Red Pyrometer Glasses," *Astrophys. J.*, **42**, 302 (1915).

[3] M. Luckiesh, "Color and Its Applications," **1915**, D. Van Nostrand Co., New York.

phenomenon can be made spectrophotometrically and in parallel with chemical investigations.

In order to eliminate the annoyance of large color-differences in determining the transmission-factors at different temperatures, a given specimen was cut into two pieces and one was kept at a temperature of 30° C, while the temperature of the other was altered gradually from this temperature to 350° C. The transmission-factor of a colored glass, of course, varies with the illuminant so that such a value is indefinite unless the illuminant is specified. In this preliminary account it appears sufficient to state that the illuminants used were gas-filled Mazda lamps operating at normal voltage. A continuous check on the constancy of the light-sources and of the transmission-factors of the optical paths was made possible by removing the two colored glasses from the optical paths momentarily without altering the temperature conditions. The relative transmission-factors of the two pieces of the given specimen were measured throughout the range of temperature indicated and the results for ten commercial specimens are given in the diagram and in the table. (Fig. 1 and Table 1.)

TABLE 1.

Specimen.	Principal coloring element.	Color.		Relative transmission-factors at various temperatures (centigrade).				
		Cold.	Hot.	30°.	100°.	200°.	300°.	350°.
1	Copper	Medium red	.....	100	97	92	87	84
2	Cobalt	Light blue	.....	100	101	104	107	108
3	Cobalt	Deep violet	Deep blue	No appreciable change				
4	Gold	Pink	Violet	100	99	96	94	93
5	Copper	Blue-green	Yellow-green	100	98	94	87	82
6	Manganese	Purple	Blue-violet	100	97	94	91	90
7	.....	Lemon-yellow	Orange	100	94	84	75	71
8	.....	Dull yellow	.....	100	98	94	91	90
9	Copper	Deep red	.....	100	86	67	50	42
10	Chromium	Yellowish green	Yellow	100	95	84	72	67

No color-difference was encountered during the measurements except that due to a change in the spectral transmission charac-

teristic of the heated specimen. This color-difference became very marked for specimens 5 and 10. The transmission-factor of the hotter piece is given in terms of that of the colder piece of the same specimen at the various temperatures—that is, the transmission-factors as given are relative values and not absolute. The color of a specimen at the highest temperature is given as compared with that of a piece of the same specimen at 30° C, the change being sufficient to be readily described in

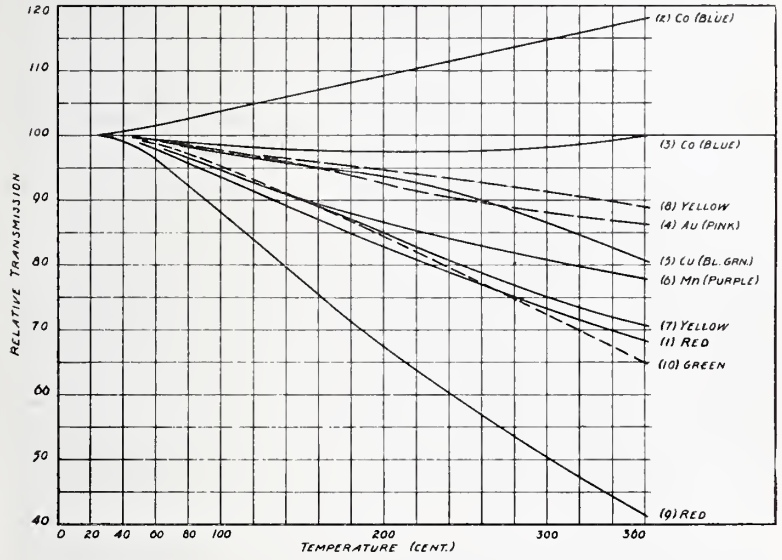


FIG. 1.

terms of our ordinary indefinite terminology. All the glasses excepting the two containing cobalt decreased in transmission-factor as the temperature increased, and in some cases this decrease in transmission-factor was very large. The curves obtained by plotting temperature and relative transmission-factor are, in general, approximately straight lines indicating that throughout this range of temperature the transmission-factor changes approximately proportionally with the temperature for the specimens used. Owing to the relatively slight change in hue in the red end of the spectrum, the red glasses 1 and 9 did not change

appreciably in color when heated, notwithstanding large decreases in their transmission-factors.

This preliminary study indicates an interesting field for careful research which might throw more light upon the question, "How are glasses colored?" It is the hope of the author to carry this investigation further. In the meantime, so far as general conclusions can be drawn from this series of glasses which covers a wide range, it appears that the transmission-factor of colored glasses generally is a function of the temperature within the range investigated. It will be noted that the highest temperature studied is below that at which the glass becomes self-luminous or plastic. It will be of interest to carry this investigation close to the melting point. The results obtained are of interest both theoretically and practically.

NELA RESEARCH LABORATORY,
CLEVELAND, OHIO,
July, 1919.

THE CONTROL OF THE SAGGER MIXTURE.

By V. S. Schory.

Manufacturers who fire wares in saggers are at times under the necessity of making changes in the sagger mixture. A common method of substituting a new clay for one previously in use is to make a visual examination of fired pieces of the various clays and from such an inspection to decide upon a new sagger mixture. This practice, depending partly on judgment based on insufficient data and partly on pure guess-work, is likely to prove expensive through loss of saggers and ware. Another method is to prepare a number of sagger mixtures in which one or more of the materials are varied and to study the saggers made of these mixtures. It is no doubt possible to judge roughly of the quality of a sagger by inspection and breaking with a hammer, but if accurate information is desired, it is necessary to select a group of say 25, 50 or 100 saggers and fire and re-fire them under standard conditions until all are broken, meanwhile keeping a record of their behavior. In this way a numerical expression for the quality of saggers from a given mixture can be obtained. This method requires considerable time and work and is not altogether inexpensive, expecially if some low-grade mixtures are made.

It is sometimes necessary to make a change in the sagger mixture on short notice, thus precluding the possibility of any such research as that mentioned above. This exigency has occurred more frequently during the last few years because of the abnormal conditions prevailing in the clay-mining industry and in transportation. The extensive re-adjustment of sources of supply, the congestion of freight, and the difficulties experienced by the clay shippers in securing the needed supply of cars, have resulted in unforeseen shortages in the clay bins. When the stock of one or more sagger clays is exhausted it may be necessary, in order to maintain the usual rate of production, to make a sagger mixture from the sagger clays remaining in stock. It

may not be possible to make a sagger of the standard quality from these materials but the problem at the moment is to produce the best sagger that it is possible to make from the clays available.

Under the stress of such circumstances it seemed desirable to devise some preliminary test that would serve as a guide in making a change in the sagger mixture. No attempt was made to formulate tests from which to absolutely predict the quality of the sagger. A great many variables affect the life of a sagger and not the least important of these are the inherent properties of the individual clays. So that, granting the feasibility of the scheme, it would require an extended research to formulate a series of tests that would give a numerical rating to any sagger mixture, applicable to all clays and combinations of clays. Instead, the purpose was to devise a preliminary test that would eliminate as much laborious experimenting as possible, but which would not supersede entirely the determination of the average life of a sagger by re-firing as mentioned above.

From the information available it is evident that the clay content of most sagger mixtures in factory practice consists of one or more dense-burning or vitrifying clays and one or more open-burning and comparatively refractory clays. This is not always the case. In some sagger mixtures the clay content is derived from a single clay or a mixture of clays, all of which resemble each other in vitrification behavior. If the clay content of a sagger mixture consists of one vitrifying and one open-burning clay, the proportions used should be such as to give the sagger satisfactory strength and refractoriness.[1] In the usual case if the content of open-burning clay is high, the resistance of the sagger to temperature changes is correspondingly great but the strength is low. In the extreme case the sagger is often of little value because of brittleness in the cold or because of deformation under load in the fire.

The relative importance of these two properties will vary in different factories. Some users require great resistance to changes of temperature; in other cases mechanical strength is more important. For any one use there is the desirable mean in which

[1] G. H. Brown, "Notes on Sagger Clays and Mixtures," *J. Am. Ceram. Soc.*, 1, 716 (1918).

the properties are developed in proportion to their relative importance. An effort was made to locate this mean, approximately, by determining the degree of vitrification of satisfactory sagger mixtures.

A number of sagger mixtures that have given satisfactory results in factory practice were selected. The clay content of four of these consisted of clays that vitrified at or before cone 11, combined with clays which after firing to cone 11 absorbed between 6 and 10 per cent of water; the clay content of one mixture consisted of two clays that burned to low absorption but were not vitreous at this heat. Small batches of these mixes were weighed up, the grog being omitted in order to avoid any error that might arise from variations in the absorption of the grog particles. The batches were blunged, brought to the plastic condition, formed into bars, dried, and fired to cone 11, the regular sagger-kiln heat. The absorptions of the bars were determined by the increase in weight after boiling for three hours and soaking twenty-four hours in water. The absorptions in all cases were between 2.5 and 4 per cent.

Similar tests were made using the clay content of sagger mixtures that had proven unsatisfactory in use. These mixtures consisted of the same clays as were used in some of the satisfactory mixtures but the clays were combined in different proportions. Fired bars of these mixtures showed absorptions either greater than 4.5 per cent or less than 1.5 per cent.

The number of mixtures studied was too small to permit of drawing positive conclusions. However, within the limits of the study, no exceptions were found to the rule that mixtures of clays showing absorptions between 2.5 per cent and 4 per cent are superior to mixtures of the same clays having absorptions much less than 2.5 per cent or greater than 4 per cent. These data classified the sagger batches that had been used in an emergency when there was no time for experimenting and had later proven inferior in quality. It was, therefore, decided to base future work on these results until exceptions to the rule occurred. By experiment the proportions of each two sagger clays that gave mixtures burning to an absorption of approximately 3.25 per cent

were determined. From these data a sagger mixture is planned immediately in case the supply of one clay is exhausted.

The absorption determination is also used as a preliminary to making a study of a new sagger clay. The new clay is mixed with a standard open-burning clay or a standard vitrifying clay in such proportions that the absorption of the resulting pieces is approximately 3.25 per cent. This mixture is then used as the clay content of a sagger batch and the average life of a sagger is determined by re-firing a given number until all are broken. This figure is taken as a measure of the value of the new clay.

This method of control of the sagger mixture is very simple and has proven useful in factory practice. It is presented for the consideration of other users of saggers who may at times be under the necessity of changing their sagger mixtures. The degree of vitrification represented by the figures for absorption is not given as a generalization. Individual users may secure better results with a greater or lesser degree of vitrification than that mentioned above.

Standard Sanitary Mfg. Co.,
Tiffin, Ohio.

CERAMIC ABSTRACTS.

General.

Rapid method of determining magnesium. F. W. Bruckmiller. *The Chemist-Analyst, J. T. Baker Chemical Co.*, p. 7, Jan. (1919).—Remove the silica and calcium in the usual manner and after evaporating to dryness, to get rid of the excess ammonium salts and filtering, proceed as follows: Make up the filtrate to at least 100 cc. volume and make distinctly alkaline with NH_4OH. Cool by placing the beaker into ice water and then add an excess of microcosmic salt solution. Stir vigorously and for some time until the magnesium has all come down and then add 10 cc. of NH_4OH. The more the stirring the better will the precipitate settle and the sooner will it be ready to filter. Filter through a suitable filter paper and wash by decantation several times using 3 per cent ammonia water. It is not necessary to wash the beaker clean, for the contents of the filter paper are washed by means of hot water into the beaker in which the precipitation took place and the whole solution boiled until the ammonia is driven off. Methyl orange indicator is added and the solution titrated with a standard solution of HCl. The strength of the solution used will depend upon the quantity of magnesium present. The following factors will convert cubic centimeters into grams of magnesium:

1 cc. 0.10 *N* HCl	0.00120 gm. Mg
1 cc. 0.05 *N* HCl	0.00060 gm. Mg
1 cc. 0.02 *N* HCl	0.00025 gm. Mg

C. Treischel

Notes on the perchloric acid method for potash in cement materials. W. B. Newberry. *The Chemist-Analyst*, pp. 8–11, Jan. (1919).—*For total potash:* Grind 1/2 gram sample (flue dust, shale, etc.) with 1/2 gram ammonium chloride in an agate mortar. Add 4 grams of pure calcium carbonate and grind thoroughly together. Put a little calcium carbonate in the bottom of a J. Lawrence Smith crucible, add the charge with 1 gram calcium carbonate on top, cover and heat with a low flame till there is no further odor of ammonia (15 minutes). The heat must not be high enough to permit the escape of visible fumes. Then raise the heat to moderate redness for 45 minutes, turning crucible occasionally. Cool, dissolve the melt in a little water, carefully rinsing the crucible. Slake on water bath, breaking up lumps. Filter off excess calcium carbonate, wash and make filtrate strongly acid with HCl. Boil and add slight excess of barium chloride. Let stand preferably several hours. Filter off the barium sulphate, wash, heat the filtrate to boiling and add ammonium carbonate until no further precipitate forms. Add a few drops of ammonium oxalate, filter and wash. Evapo-

rate to dryness in a platinum dish and when dry heat carefully to just below redness till all ammonium chloride fumes are gone. Cool, dissolve in 25 cc. hot water, filter off anything undissolved and add to the filtrate enough 60 per cent perchloric acid solution to convert all the alkali metals into perchlorates (ordinarily 1 cc. is enough). Evaporate until nearly dry and when heavy fumes of $HClO_4$ appear, cool, add 25 cc. hot water, another cc. perchloric acid, and evaporate again to the same point. Cool, add 25 cc. wash-alcohol (containing 1 cc. perchloric acid solution to every 300 cc.), filter on asbestos in a Gooch crucible, wash with the same alcohol, dry and weigh as $KClO_4$. In washing the precipitates it is unnecessary to wash them entirely chlorine-free; for ordinary work 3 or 4 washings are sufficient. The barium chloride, ammonium carbonate and ammonium oxalate mentioned are all used in saturated solutions.

For water soluble potash: Five grams of the sample are boiled thirty minutes with 150 cc. water in a small flask with condensing tube. Cool, make up to 250 cc., mix well and filter through a dry paper without washing. Take 50 cc. of the filtrate corresponding to one gram of the sample, acidify strongly with HCl and proceed as in the determination of total potash given above. Multiply weight of potassium perchlorate found by 33.935 for percent of K_2O in sample. C. TREISCHEL

Geological.

Ball clays of West Tennessee. ROLF A. SCHROEDER. *The Resources of Tenn.*, **9**, No. 2, April (1919).—High-grade ball clays are found in abundance in Henry and Carroll Counties and sparingly in Haywood and Madison counties. South of these counties clay strata are abundant but are less pure and contain quartz, mica and iron compounds. The best ball clays of Henry and Carroll counties are apparently associated with lignite. The clays of West Tennessee are in the form of lenses which are found in the upper portions of the Ripley sand (Upper Cretaceous) and the lower Lagrange sand (Eocene). According to Schroeder some of the more important uses of ball clay are as follows: (1) *Electric porcelain ware and insulators.* (2) *Porcelain or china.* (3) *Glass pots for optical glasses.* (4) *Sanitary ware and enamel ware.* (5) *In making "graphite crucibles, as a binder in abrasive wheels, in making green-house wares, tiling, lead pencils, chemical ware and cosmetics."* The report contains many descriptions of localities arranged by counties. Many of the clays were tested at the Ceramic Laboratory of the University of Illinois under the direction of Prof. C. W. Parmelee. The results of these tests and the descriptions of prospects and worked pits are too numerous to be considered here. The original report is especially worthy of consideration by any who may be interested in the clays of the region involved. E. D. ELSTON.

Annual report on the mineral production of Canada, 1917. Dept. Mines, Mines Branch, Ottawa (1919).—A series of tables shows the domestic production and imports of clay and clay products. In addition the production of clay products by provinces is stated. E. D. ELSTON.

Preliminary report of the mineral production of Canada, 1918. Can. Dept. Mines, Mines Branch, Ottawa, Feb. 27, 1919.—The report is interesting, in that it shows the increase or decrease in production, as the case may be, with respect to the various materials. According to this preliminary set of statistics for 1918, a decrease of $179,203 is shown for clay products.

E. D. ELSTON.

Mineral production of the United States in 1916. H. D. MCCASKEY AND MARTHA B. CLARK. U. S. Geol. Surv., *Mineral Resources, 1916.* Part I, *A*, June 28, 1919.—A brief résumé of the value of clay and clay products and the value of imports of such materials is to be noted in this publication. The mineral production by states is given in tabular form and it is also worth while to note that a statement dealing with the mineral products of the United States in 1916 in order of value is included. In this statement clay products are given fifth place and raw clay is cited as twenty-seventh in value. For further details in connection with the numerous tabulated statements the reader is referred to the original publication. E. D. ELSTON.

Magnesite: its geology, products and their uses. C. D. DOLMAN. *Am. Inst. Min. and Metal. Engrs.*, **152**, 1913 (1919).—In 1913, about 200,000 tons of magnesite were consumed in the United States, less than 3% of this amt. being produced at home. In 1918 there were produced and used in the United States 225,000 tons. The material produced and sold in the United States must contain in the crude form, no more than 3.5% SiO_2 and 2% CaO, and in the dead-burned product not more than 7% SiO_2, 4% CaO, and 8% Fe_2O_3 plus Al_2O_3. There are two principal sources of magnesite in the United States; California, and Stevens County, Washington. The California magnesite is similar to the Grecian, being amorphous and occurring in serpentine rocks. The Washington deposits are similar to the Austro-Hungarian and Canadian ores and are of the massive crystalline variety. Below are given typical analyses:

	MgO.	CaO.	CO_2.	SiO_2.	FeO.	Fe_2O_3.
Napa County, Cal.....	45.01	0.67	50.65	1.99	1.49	
Grecian magnesite.....	45.75	1.44	49.88	1.63		1.36
Stevens County, Wash.....	45.20	1.74	49.70	1.85	0.88	
Austro-Hungarian magnesite.	43.82	0.97	50.44	0.45		3.65

With a production of 110,000 tons of crude ore from June, 1918, to April, 1919, only 20%, or one ton in five, mined at the quarries of the Northwest Magnesite Co., in Washington, was waste, whereas, in the Austro-Hungarian quarries it is necessary to mine and sort five tons of crude ore to produce one ton of finished product. Thus with close sorting a product just as pure, if not more pure than the foreign material, may be obtained. Geological surveys indicate that there are 7,000,000 tons of magnesite in the Stevens

County district, and exploratory drilling may multiply this estimate many fold. The deposits occur as a replacement of lenses of dolomite in sedimentary rocks, probably of pre-Cambrian age. The greatest use for magnesite is in the basic open-hearth process of making steel. It is used in furnace bottoms, soaking pits, metal mixers, billet and bar heating furnaces, copper converters, and in many types of furnaces. The magnesite burned to 1200° C is called "calcined magnesite," that burned much harder is called "dead-burned." Ferro-magnesite is made by the careful grinding and mixing of iron ore with the raw magnesite and burning to a sintering temperature. Magnesite is now put to many uses, some of which are as follows: refractory porcelains, lining for electric furnaces, composition flooring, stucco, and other oxychloride cements, boiler and steam-pipe insulation, for the production of Mg metal for important new alloys with Al and Pb, for production of $MgSO_4$, $MgCl_2$ and $Mg(OH)_2$. F. A. KIRKPATRICK.

Refractories.

Experiments on silica brick. M. PHILIPON. *Rev. de Metal.*, **15**, 51 (1918).—Four different materials having the following compositions were used:

	1.	2.	3.	4.
SiO_2	97.20	97.00	96.30	97.60
Al_2O_3	0.78	1.82	2.40	0.95
Fe_2O_3	0.37	0.60	0.40	0.52
CaO	0.90	0.00	0.28	0.00
MgO	0.00	0.00	0.00	0.00
Ignition loss	0.60	0.50	1.00	0.80
Fusion temp. ° C	1770	1780	1790	1770

1. White quartz from the Central Plateau.
2. Normandy quartz.
3. Sandstone from Pas-de-Calais.
4. Amorphous silica from Allier.

The materials were ground and separated into portions of different size of grain, the average size of grain of each portion being used in interpreting the results. $1\frac{1}{2}\%$ of CaO was used. Water was added and the mixtures ground wet for 10 mins. and molded into ordinary-sized brick. These were dried on iron plates at 100° C and burned to 1300° C, the conditions being kept alike in all the tests.

The results obtained are shown in the following tables:

TABLE 1.

Showing Effect of Size of Grain on Compressive Strength of Brick Burned to 1300° C. Figures by Interpolation from Author's Fig. 2.

Diameter of grains in mm.	Nearest corresponding mesh of Tyler standard testing sieves.	Compressive strength. Kg. per sq. cm. 1.	2.	4.
0.05	300	250	300	210
0.06		140	230	80
0.07	200	50	150	25
0.08		23	80	5
0.09		14	55	
0.10	150	8	37	
0.12		5	20	
0.14		3	10	
0.16			5	
0.18	80		4	

No mixtures of different sizes were used.

TABLE 2.

Showing Effect of Amount of CaO on Compressive Strength of Brick after Burning to 1300° C. By Interpolation from Author's Fig. 5.

% CaO.	Crushing Strength, kg. per sq. cm. No. 1.	2.	3.
0	50	110	
0.5	172	208	147
1.0	278	270	220
1.5	310	290	253
2.0	304	287	263
2.5	262	252	257

TABLE 3.

Effect of Amount of Water on Strength of Brick of Material Passing 200-Mesh Sieve.

% Water.	Compressive strength after drying. Kg. per sq. cm.	Compressive strength after burning to 1300° C. Kg. per sq. cm.
15	12	180
16	13	195
17	15	190
18	15	220
19	17	250
20	19	265
21	20	270

TABLE 4.

Effect of Amount of Lime on Fusion Temperature.

% CaO.	0.	0.5.	1.0.	2.5.
Quartz III	1790	1780	1750	1740
Quartz I	1770		1760	1730

TABLE 5.

Effect of Size of Grain and Amount of Lime upon Various Properties.

		Compressive strength. Kg. per sq. cm.	Per cent expansion.	Porosity.
Quartz 1	A	270	1.2	18.0
	B	285	1.4	18.6
	C	243	1.5	17.7
	D	194	1.6	16.2
Quartz 2	A	242	1.1	22.0
	B	223	1.1	19.3
	C	225	1.2	18.5
	D	205	1.3	17.0
Quartz 3	A	200	1.3	21.0
	B	228	1.5	19.8
	C	175	1.4	19.0
	D	165	1.5	18.2
Quartz 4	A	194	1.4	20.5
	B	171	1.5	19.7
	C	155	1.6	17.8
	D	138	1.8	15.9

	Per cent passing 200 mesh.	Per cent grains 2 to 8 mm.	Per cent CaO.
Series A	60	40	1.2
B	50	50	1.0
C	40	60	0.8
D	30	70	0.6

F. A. KIRKPATRICK.

Cement and Lime.

Cements producing quick-hardening concrete. P. H. BATES. *Concrete* (Cement Mill Section), 15, 9 (1919). *Presented at the 1919 meeting of the American Society for Testing Materials.*—In the course of investigations which the Pittsburgh branch of the Bureau of Standards has been conducting in portland cement and related problems, there has been produced a quick-setting sorel-cement concrete which obtains the greater part of its strength in 24 hours. Tests were first made of mortar having the composition of so-called "composition flooring" much used in office buildings, passenger cars, and other places. Such a mortar has been successfully used as a stucco. Cali-

fornia magnesite was used. The specimens were composed of 50% magnesia, 12% asbestos, 10% sawdust, 22% ground sand, and 6% iron oxide, all by weight. This mixture was gauged with 52% of a solution of magnesium chloride having a gravity of 1.885 (about 93° Baume). Analyses are as follows:

	Oxide, per cent.	Chloride, per cent.
SiO_2	5.14	
Al_2O_3	0.78	
Fe_2O_3	0.57	
CaO	5.69	0.20
MgO	85.01	9.24
K_2O		0.17
Na_2O		0.70
Cl		16.54
Ignition loss	2.86	

Tension specimens were of the forms specified in the A. S. T. M. "Standard Specifications and Tests for Portland Cement." Compression specimens were 2 inch by 4 inch cylinders. The following strengths in pounds per square inch were obtained:

	Specimen.	24 hours.	7 days.	28 days.	90 days.
A	Tension	531	841	1087	1034
	Compression	2240	3795	5300	4340
B	Tension	576	900	935	1049
	Compression	2900	4240	5300	5370

This mortar was used in making a concrete. The strength of this concrete and also the compressive strength developed by a portland cement concrete using the same proportions of the same aggregates are given in Table 1.

As the magnesia weighed but 51 lb. per cu. ft., the actual percentage by weight of the magnesia in any one concrete was decidedly less than the percentage of Portland cement. For this reason the 1 : 6 concrete was proportioned both by weight and by vol. when the magnesia was used as the cementing material. All specimens were stored in the air until broken. The gravel used was obtained from the Allegheny River and was screened between a $1/8''$ and $1\,1/2''$ screen. It contained an excessive amt. of the finer sizes. The limestone was a rather soft stone screened between the $1/4''$ and $1''$ screens. The "coarse sand" aggregate was sand from the Allegheny River screened between the $1/8''$ and $1/4''$ screens. As the information sought had to do alone with early strengths, specimens were made for breaking at 24 hrs., 48 hrs. and 7 days, in the case of those made with magnesia, and 48 hrs. and 7 days when Portland cement was used. The results show that the Sorel-cement concretes, with but two exceptions, have a strength at 48 hrs. greater than that obtained by the use of Portland cement in 7 days. However, it is also seen that there is in general but little increase in the strength of the

TABLE I.

Comparison of Compressive Strengths of Sorel and Portland Cement Coneretes.

6″ × 12″ Cylinders. Strengths in Pounds per Square Ineh.

Proportions by volume.	Percentage of cement by weight.		24 Hours.		48 Hours.		7 Days.		Per cent H_2O in Portland-cement concrete.	Per cent $MgCl_2$ in Sorel cement concrete.
	Portland.	MgO.	Portland.	MgO.	Portland.	MgO.	Portland.	MgO.		
Gravel aggregate.										
1 : 0.5 : 1.5	33.3	20.3	....	1740	1570	3490	2965	2910	12.1	14.7
1 : 1.0 : 3.0	20.0	11.3	....	1705	1115	2260	2020	2850	7.8	9.5
1 : 1.5 : 4.5	14.3	7.8	....	1755	680	2145	1430	2240	7.9	9.8
[a] 1 : 1.5 : 4.5	....	14.3	....	2880	....	3315	....	2720	....	12.1
Limestone aggregate.										
1 : 2/3 : 1 1/3	35.2	21.8	....	2985	2260	3990	3930	4320	12.2	18.5
1 : 1 1/3 : 2 2/3	21.4	12.2	....	1940	1340	2805	2990	2970	9.9	13.7
1 : 2 : 4	15.4	8.5	....	1935	655	2330	1755	2480	9.5	12.8
[a] 1 : 2 : 4	....	15.4	....	3270	....	4020	....	4320	....	13.7
Coarse-Sand aggregate.										
1 : 0.5 : 1.5	34.0	20.8	....	2060	1715	2570	3270	2735	13.2	20.0
1 : 1.0 : 3.0	20.6	11.6	....	1910	545	2225	1635	2430	11.4	16.0
1 : 1.5 : 4.5	14.7	8.0	....	935	325	1695	985	1870	11.4	14.7
[a] 1 : 1.5 : 4.5	....	14.7	....	2355	....	3260	....	3190	....	15.7

NOTE.—The pereentage by weight of MgO in 1 : 0.5 : 1.5 and 1 : 2/3 : 1 1/3 is but very little greater than the pereentage by weight of Portland cement in 1 : 1.0 : 3.0 and 1 : 1 1/3 : 2 2/3, respeetively.

[a] MgO proportioned by weight, making weight percentage of MgO equal weight pereentage Portland eement in 1 : 1.5 : 4.5 and 1 : 2 : 4.

former between the 48-hr. period and the 7-day period. The results obtained at all periods from the Sorel cement concrete proportioned by wt. are generally higher than those obtained when the proportioning was done by vol., regardless of what the proportions were. This clearly shows that for such a light material the proportions should be decidedly different from those generally used with Portland cement. It would seem as if too much magnesia was used in the rich concretes and too little in the lean ones when the proportioning was carried out by vol.

Attention should be called to the difference in the manner in which the hardening of the two concretes takes place. In a Portland-cement concrete, the cement hardens by the action of the added water. During the process of hardening it is immersed in an atmosphere containing in a greater or less degree one of the essential elements of the process, that is, water, either as a liquid or vapor. On the other hand, in the Sorel cement concrete the hardening takes place as a result of the action of the chloride on the oxide. The chloride is always essential but any deficiency, as for instance the use of a too dilute solution, or any marked excess, is injurious. Therefore, not only the ratios between the aggregate and the magnesia must be studied, but also the ratio between the chloride and the magnesia.

While the results do show the lack of proper proportioning of both the magnesia and the chloride, yet these poorly proportioned concretes show in 24 hrs. as high strengths as the more properly proportioned Portland cement concretes show in seven days. Another point should be remembered in regard to these Sorel cement concretes, that is, they cannot be frozen. Temps. below freezing will retard the hardening to a degree, but the freezing point of the solution is so low that no care need be taken to prevent damage from low temps. The remainder of the discussion concerns the properties of calcium-aluminate cements and concretes, previously published in THIS JOURNAL, **1**, 679 (1918). F. A. KIRKPATRICK.

New wet process plant of Nebraska Cement Company. D. C. FINDLAY. *Concrete* (Concrete Cement Mill Section), **15**, 1 (1919).—The plant, located at Superior, Neb., has a maximum capacity of 3,000 barrels per day. All buildings, storage tanks, slurry elevators, and every unit that could be advantageously built so, were made of reinforced concrete. Soft, yellow limestone and blue clay are dug by steam shovel and hauled by locomotives $2^1/_2$ miles to the plant. The materials are reduced by a 38″ by 60″ Fairmont crusher, then ground into slurry in "Compeb" ball mills. The slurry is discharged into any of three reinforced concrete correcting basins holding about 200 barrels of slurry. The latter is analyzed and drawn from the basins in proper proportions into a Smidth type mixing basin of 1,000 barrels capacity. When the resulting mix is absolutely correct it is drawn off into any of three storage basins, two of which are 26 feet in depth and hold 1750 barrels each, and one 16 feet in depth holding 1,000 barrels. The slurry is then elevated into storage basins above the kilns and fed by means of Link-Belt "S" type slurry feeders. The three rotary kilns are 8 feet in diameter and 200 feet long.

The kind of inside lining is not stated. The kilns are fired with powdered coal. The clinker passes into two 60-foot rotary coolers, then into four storage bins 26 feet in diameter and 45 feet high. At this point, gypsum is added and the product ground in five 57-inch Fuller mills and is then elevated to the storage bins. There are 10 bins, each 26 feet in diameter and 80 feet high with walls 7 feet thick, the total capacity being 100,000 barrels. The entire plant is motor driven, there being in all some 50 motors in service. The power plant is equipped with a 1500 kw. Westinghouse steam turbine with Le Blanc condenser, and a 2000 kw. Allis-Chalmers turbine and a jet condenser. Steam is supplied by four 450 h. p. water tube boilers equipped with Taylor forced-draft stokers. F. A. KIRKPATRICK.

CERAMIC PATENTS.

G. E. MIDDLETON, ABSTRACTOR

Truck for tunnel kilns. C. H. ZWERMANN. U. S. 1,306,160, June 10, 1919. The frame of this truck is of channel irons. Upon this frame is a sheet metal platform upon which rests a series of transverse I-beams embedded in kieselguhr. The platform proper which rests upon these I-beams is composed of overlapping, interlocking fire-clay blocks. As these blocks become worn they can be replaced.

Tunnel kiln. C. H. ZWERMANN. U. S. 1,306,161, June 10, 1919. This is a twin-tunnel muffle kiln, the two tunnels being parallel and having a common inner wall. The ware in one passes in a direction opposite to that of the ware in the other, thus effecting a mutual cooling and preheating through the medium of a specially designed central wall in the end portions of the kiln. The combustion chambers extend longitudinally of the tunnels, the walls thereof being of relatively thin plates of carborundum capable of withstanding great heat and at the same time readily transmitting it. The ring is longitudinal. The fuel is either oil or gas.

Brick-mold-handling apparatus. I. C. FREY. U. S. 1,306,477, June 10, 1919. This apparatus comprises synchronously operating, divergently moving conveyors for separating a mold from its contents, means for moving the emptied mold to a sander by which it is coated with a regulated quantity of sand, turning the sanded mold to the upright or filling position and automatically returning the mold to the filling chamber of a brick machine.

Glass-matting process. T. UEDO. U. S. 1,306,505, June 10, 1919. A paste is made comprising active ingredients, such as basic alkali silicates, basic lead silicates, lead oxides, plumbates and lead borates; inert ingredients, such as pulverized highly refractory material, for example, burned porcelain, burned stoneware, fire clays, quartz, etc.; and a binder, such as boiled starch, gum, glue, etc. This is painted in appropriate design upon the glass surface to be matted and the whole placed in a furnace and heated at a temperature higher than the softening point of the glass for a time sufficiently long to

effect a fluxing action upon the glass by the active ingredient. The glass is then cooled and treated with any ordinary strong mineral acid. During the heating the active ingredients combine chemically with a thin surface layer of the softened glass forming a new combination, which, on account of the presence of the inert materials, can be easily decomposed and removed by an acid, thus effecting the matting. The inert materials function principally to prevent the active materials and the reaction products from sticking so fast to the glass surface as to make their decomposition by the acid difficult.

Process of drawing glass cylinders. W. WESTBURY. U. S. 1,306,651, June 10, 1919. During the elevation of the bait a complete air circuit is maintained within the cylinder being drawn, said circuit including downwardly and upwardly flowing air currents, the upward current coming into contact with the surface of the cylinder.

Glass-stowing tool. J. A. BECHTEL. U. S. 1,306,993, June 17, 1919. A reversible stowing tool having its blade or that portion which comes in contact with the glass of greater length than the linear dimension of that edge of the glass plate with which the tool contacts.

Glass-delivering and melting apparatus. E. ROIRANT. U. S. 1,307,150, June 17, 1919. Molten glass is collected from a furnace by means of a receptacle which is swung from position under the delivery end of the furnace to the gathering device and back again. During this operation the glass in the receptacle may be kept molten by passing an electric current through it.

Magnesia brick and method of making the same. H. H. HANSON. U. S. 1,307,197, June 17, 1919. Calcined magnesite or magnesia, 83%, is mixed with 17% fireclay, then a small amount of sodium silicate and water is added and the whole thoroughly mixed in a pug mill. It is then formed into bricks subjected to a pressure of six tons per square inch, and then subjected to 1600° C. The product is said to be a practically non-porous, basic brick expanding very little when heated to high temperatures.

Splitting buck for glass cylinders. J. MURPHY. U. S. 1,307,209, June 17, 1919. This buck has two sets of supports, one consisting of flexible strips of fabric for holding the cylinder during splitting and another consisting of two sets of anti-friction rollers, one set at each end of the buck, which may be raised to lift the cylinder from the first-mentioned supports and to permit of its being rotated for inspection preparatory to splitting.

Apparatus for the manufacture of plate glass. H. K. HITCHCOCK. U. S. 1,307,357, June 24, 1919. By means of this apparatus the use of tank furnace glass for the manufacture of plate glass is said to be rendered practical and economical. The operation of the apparatus by which the glass is caused to flow from the tank to the casting-table is automatically controlled by the movement of the table itself below the glass delivery spout.

Window-glass-blowing head. W. E. STANDLEY. U. S. 1,307,381, June 24, 1919. The construction of this head enables the flow of air to and from the hollow bait to be regulated to maintain a uniform pressure within the hollow cylinder.

Machine for fire-finishing tumblers and other glass articles. H. H. PITT. U. S. 1,307,453, June 24, 1919. Means are provided for passing water, steam, air or other cooling medium through the plugs or stands which support the hollow glass articles being fire-finished, thereby cooling the interiors of the articles and preventing their sticking to the plugs.

Glass furnace. F. L. O. WADSWORTH. U. S. 1,307,527, June 24, 1919. This furnace is designed to deliver in an upward direction a constant stream of molten glass under a gravity head in such condition that open-bottomed receptacles may be successively brought into register with the delivery opening to receive comparatively small quantities of molten glass and when withdrawn the flow of molten glass will continue and will fall away from the orifice.

Heat insulating composition. F. A. HEADSON. U. S. 1,307,548, June 24, 1919. Calcined diatomaceous earth, 75%, finely divided or fibrous asbestos either calcined or raw, preferably the former, 25%, either with or without a binder of refractory or bonding clay amounting to 3% to 10% of the combined diatomaceous earth and asbestos. This composition is molded in any desired shape and burned at from 2000° F to 3000° F for six hours.

U. S. 1,307,549 discloses substantially the above.

Utilization of cement-kiln dust. R. J. NESTELL. U. S. 1,307,920, June 24, 1919. Cement-kiln dust, which sometimes contains as high as 13% K_2O, nearly all water soluble, is rendered suitable for use as a crude fertilizer by first mixing it with water and then passing hot kiln gases containing CO_2 through the mixture in such a manner as to increase the solubility of the potassium compound and to carbonate any free lime present.

Glass-drawing apparatus. W. WESTBURY. U. S. 1,307,943, June 24, 1919. By the use of this apparatus a cylinder may be formed from a charge of glass taken from the melting furnace, drawn from a pot which closes or seals the drawing opening and in which the charge of glass is exposed to atmospheric cooling while protected from the heat of the furnace.

U. S. 1,307,946 discloses substantially the above. The claims are drawn to the process.

Brick-carrier. W. H. ALLEN. U. S. 1,308,021, July 1, 1919. By means of this device a number of bricks can be transferred from a stack or kiln to a car or *vice versa*, thereby eliminating manual handling.

Tile press. F. B. YINGLING. U. S. 1,308,213, July 1, 1919. A toggle-operated dry-press having a reciprocating charging hopper comprising means for agitating the clay therein. The surfaces of the dies are automatically cleaned by means of a brush supported by the charging hopper.

Glass-bait. G. C. DEBAY. U. S. 1,308,278, July 1, 1919. The necessity for submerging this bait in the molten glass to start the draw is avoided by the use of a suction means which draws the glass up into an annular cavity in the bottom of the bait. Means are also provided for relieving the strain on the glass cylinder due to cooling and contracting.

Glass-carrying rod for glass-annealing leers. J. W. CRUICKSHANK. U. S. 1,308,337, July 1, 1919. This carrying rod or lifting bar is designed to be of low conductivity and thus avoid either chilling or heating the sheet of glass being annealed. The bar is made of two angle irons riveted together and carrying between them a strip of asbestos projecting beyond the working faces of the irons.

Glass-flattening oven. H. E. DEVAUGHN. U. S. 1,308,341, July 1, 1919. The driving mechanism of this flattening oven is outside and above it and the rotary wheel is suspended from a support located at a point such that the temperature of the oven can have no detrimental effect upon the operation. The making of repairs is thus expedited.

Process and apparatus for making glass plate letters. A. W. GAST. U. S. 1,308,408, July 1, 1919. Glass-plate blanks of the desired size are passed through a longitudinal heating chamber upon suitable supports and when attaining the proper temperature are impressed by means of a heated pneumatic die. The formed blank is then passed through the remaining length of the longitudinal chamber and slowly cooled.

Treatment of clays and earths. F. LANGFORD. U. S. 1,308,429, July 1, 1919. This process relates to the production of aluminum and aluminum compounds and comprises providing a wet mixture of comminuted wood of the California Redwood or *Sequoia sempervirens* together with the earthy material, clay or ore to be treated, adding to this mixture sulphur-oxygen compounds to produce a solvent solution containing H_2SO_4 to take up the undesirable impurities without dissolving the aluminum content of the mixture, separating the insoluble aluminum content from the solution, treating the insoluble aluminum residue with H_2SO_4 to form a solution of sulphates and an insoluble residue, separating the solution of sulphates from the insoluble residue and roasting the sulphates to provide the sulphur compounds for the treatment of the mixture of wood and earthy materials.

Protecting refractory furnace-linings. H. T. CHAPPELL. U. S. 1,308,481, July 1, 1919. Refractory furnace linings containing silica are protected from fusion and disintegration by supplying finely-divided calcined and leached alunite to such linings when they are at such a temperature as to be soft and partly fused.

Process of molding lenses. E. D. Tillyer and H. H. Styll. U. S. 1,308,820, July 8, 1919. Ophthalmic lenses are formed by first heating the glass blank, moistening the surfaces of the mold, placing the glass blanks between the mold sections while in a heated condition and then molding. The vapor films thus formed act as cushions preserving the fire polish of the glass while it is being shaped.

Process of treating lime. C. Catlett. U. S. 1,308,931, July 8, 1919. Lime is slacked, preparatory to making lime plaster or mortar, by adding sufficient water and up to about 5 per cent of an oxychloride of calcium, magnesium or zinc, or mixtures thereof. The resulting plaster is said to be hard, tough and relatively free from any tendency to blister or check after application.

Cement composition and method of preparing it. C. Catlett. U. S. 1,308,932, July 8, 1919. Relatively high, early-time strengths are developed in Portland cement mortars or concretes by the addition of not substantially more than 10 per cent of an oxychloride of calcium, magnesium or zinc, or mixtures thereof.

Continuous tunnel kiln. C. H. Zwermann. U. S. 1,308,973, July 8, 1919. A twin tunnel kiln, the two tunnels having a common inner wall. The ware passes in opposite directions in the two tunnels. The construction and operation of this kiln is somewhat similar to that of U. S. 1,306,161. The method of firing and controlling the draft is, however, quite different, this being an open fire kiln and similar to U. S. 1,275,352.

Sandless concrete. C. H. Light. U. S. 1,309,038, July 8, 1919. A sandless concrete which consists of sawdust 8 parts, cement 4 parts, plaster of Paris 1 part, and lime 1 part.

Glass blower. E. O. Whitley and E. L. Knowlton. U. S. 1,309,166, July 8, 1919. A glass-blower designed so that a plurality of tumblers may be drawn and blown at the same operation.

Glass apparatus. R. W. Hilton. U. S. 1,309,199, July 8, 1919. Means are provided in a glass furnace for supporting and shifting the pots in such a manner that when one pot is in position for the drawing of the cylinder, another pot will be in position for draining.

Kiln-furnace. R. W. Menk. U. S. 1,309,213, July 8, 1919. This furnace is shown in use on a downdraft kiln. The furnace consists of two parts, one without the kiln wall proper, the other within the bag wall. After the fire is started on the outer grate bars, the burning fuel is pushed back on grate bars within the bag walls. Within the outer furnace there is a removable corrugated, fire-clay liner tapering downwardly and outwardly so designed to prevent any "bridging" of the fuel. The draft enters above the fuel bed in the outer furnace and passes downwardly therethrough.

Process of drawing glass. J. A. CHAMBERS. U. S. 1,309,274, July 8, 1919. After a cylinder is drawn and removed a stump of hardened glass remains in the drawing ring. This patentee remelts this preparatory to a second draw by pouring molten glass through the drawing ring, this molten glass being of a higher temperature than the glass in the drawing part of the tank.

Concrete. A. MARKS. U. S. 1,309,296, July 8, 1919. A concrete consisting of Portland cement 2 parts, sawdust 1 part, sodium silicate solution sufficient to dampen and a calcium chloride solution or some easily ionized acid in such quantity that there shall be a slight excess of sodium silicate.

NOTE.—Copies of the above patents may be secured for five cents each by application to the Commissioner of Patents, Washington, D. C.

ACTIVITIES OF THE SOCIETY.

The following program will be presented on Ceramic Day, Wednesday, September 24th, at the Fifth National Exposition of Chemical Industries, Chicago, Illinois, week of September 22nd:

10:00 A.M.

"The American Ceramic Society—Past, Present and Future," Charles F. Binns.

"Buy on Analysis," Alexander Silverman.

"Superior Refractories," Ross C. Purdy.

"The Making of Pottery," Frederick H. Rhead.

"Some Aspects of Scientific Research in Relation to the Glass Industry," E. W. Washburn.

2:30 P.M.

"General Types of Optical Glass," Robert J. Montgomery.

"Brick and Tile," Douglas F. Stevens.

"The Application of Scientific Methods to Ceramic Research," A. V. Bleininger.

"The Manufacture of Optical Glass," J. C. Hostetter.

"Enameling Technology," R. R. Danielson.

"Fused Sillimanite Products," A. Malinovszky.

8:00 P.M.

Three films on ceramic subjects will be included in the motion pictures presented at the evening session.

NOTICES OF GLASS DIVISION.

Mr. Kerr has appointed the following committeee to be known as the "Central Committee of the Glass Division" to hold office until the next annual meeting of the Society: Mr. W. M. Clark, *Chairman*, Dr. S. R. Scholes, Dr. E. W. Washburn, Dr. J. C. Hostetter, Mr. R. L. Frink, Mr. F. A. Kirkpatrick, and Mr. C. O. Grafton. The duties of this Committee will be to perfect the organization of the Glass Division and to take action on such matters as may be referred to Committees.

The Society of Glass Technology of England has accepted the invitation of the Glass Division to be present at our Summer meeting in 1920. Dr. W. E. S. Turner, Secretary of that Society, is expecting to visit the States during September.

A meeting of the Glass Division was held in Cleveland on August 7th, during the Summer Session of the Society. Twenty members were present. It was voted that "Questions concerning every-day glass practice be submitted to the Central Committee and that it be instructed to hand these to men who in their minds are best able to discuss them, and that the discussions be published in the JOURNAL."

The following changes of address are noted:

C. C. Rand, Nela Park, Cleveland, Ohio.

J. C. Hostetter, Corning Glass Works, Corning, N. Y.

E. W. TILLOTSON, *Secretary*,
Glass Division

AMERICAN CERAMIC SOCIETY.

Acquisition of New Members during August, 1919.

Resident Associate.

Bloxsom, John J., Brookville Glass and Tile Co., Brookville, Pa.
Blumenthal, George, Jr., Bureau of Standards, Pittsburgh, Pa.
Brann, Albert, Ph.D., 220 Springdale Rd., East Orange, N. J.
Garrod, Fred. B., Owens Bottle Co., Toledo, Ohio.
Goebel, J., Jr., S. Highland Ave., Nyack, N. Y.
Hintze, T. Forsyth, 120 Liberty St., New York City.
Hollingsworth, C. M., Ohio Valley Clay Co., Steubenville, Ohio.
Holmes, J. C., Sandusky Bulb Works, Sandusky, Ohio.
Koerner, Walter A., Edison Lamp Works, Harrison, N. J.
Little, E. C., Laclede-Christy Clay Products Co., 1366 Peoples Gas Bldg., Chicago, Illinois.
Myers, Elmer E., Brookville Glass and Tile Co., Brookville, Pa.
Roberts, Jane E., Librarian, State University of Iowa, Iowa City, Iowa.
Shaw, L. I., Ph.D., Assistant Chief Chemist, Bureau of Mines, Washington, D. C.

Foreign Associate.

Berland, Louis, 10 Grande Rue, Villejuif, Seine, France.

JOURNAL
OF THE
AMERICAN CERAMIC SOCIETY

A monthly journal devoted to the arts and sciences related to the silicate industries.

Vol. 2 October, 1919 No. 10

EDITORIALS.

CERAMIC PRODUCTS AT THE FIFTH NATIONAL EXPOSITION OF CHEMICAL INDUSTRIES, CHICAGO, ILL., SEPT. 22–27, 1919.

The extensive and varied display of ceramic products at the Exposition demonstrated how essential the silicate industry is to chemical work. There were only a few products not previously exhibited, for the American manufacturer for the past year or so has been supplying practically everything along this line that is needed. The exhibit showed that they have been consolidating their position by extending the application of their wares, standardizing on designs and shapes and improving the quality. In some cases the increased size and intricacy of design now being made is a very decided advance in the art.

The ceramic exhibits were more numerous than those of last year. There were two exhibits on Insulating Materials, five on Chemical Stoneware, four on Enameled Ware, three on Refractories, five on Chemical Porcelain, two on Optical Glass, and seven on Chemical Glassware.

Chemical Stoneware.

New designs and shapes of acid-proof stoneware have been required in the manufacture of dyes, acids and other chemical products. The success with which the manufacturers have met the problems of shaping and molding the ware was shown by the exhibits of Maurice A. Knight, General Ceramics Company, B. Mifflin Hood Brick Company, G. H. Elmore, and the United

States Stoneware Company. Spiral coils several feet high, sharply threaded spigots, large tanks, ground valve and valve seats, equal to those of polished steel, are examples of the difficult shapes molded and the quality of the finished product. When hit with a hammer, this ware shows the strength and "ring" of cast iron.

Most of the products are glazed, although this is a choice with the buyer, as the ware is thoroughly vitrified and the acid-proof qualities are not dependent upon the glaze. Twelve different bodies are used by manufacturers to meet the special requirements in use to which the ware is put. Attempts are being made by manufacturers to standardize shapes and sizes of ware, where possible, which should be beneficial to the buyer as well as to the manufacturer of stoneware.

Enameled Wares.

Enameled wares formed a good share of the ceramic exhibits, especially acid-proof cast iron products. The exhibitors were: The Pfaudler Company, The Elyria Enameled Products Company, Vitreous Enameling Company, and J. L. Mott Iron Works. This branch of the ceramic industry has extended its products into fields where the contact of materials with metal is harmful to the finished product, such as in the manufacture of candy and dyes. There is hardly an industry where the operations of mixing and evaporating are used that is not adopting this ware. As in the stoneware industry, advancements have been made largely in the increased size of the product and its shape. However, this advancement deserves much credit.

Refractories.

Refractory products and materials were shown by the Carborundum Company, The Norton Company and The Denver Fire Clay Company.

The exhibit of The Carborundum Company included carbofrax and refrax brick, tile, muffles, tubes, carborundum refractory-cement, carbofrax saggers, fire sand, silicon metal, carborundum and aloxite wheels. The carbofrax saggers are a new

product. They have a high heat conductivity and a low coefficient of expansion, thus giving them a life many times greater than that of ordinary saggers. Two saggers on exhibition were said to have stood 211 burns to cone 13 under a load of 45 pounds. Two grades are being made, one for use above and the other below cone 12. To prevent discoloration of the ware burned in them, glazing is resorted to.

The Norton Company exhibit showed the alundum and crystolon refractories, laboratory ware, and refractory cement. Samples of ignition capsules, combustion tubes, filtering cones, pyrometer tubes and alundum and crystolon linings for electric furnaces made the exhibit an interesting one. A sample of electrically fused or sintered magnesia was worthy of notice. This product is new and has not yet been fully developed. Further investigations along this line will undoubtedly develop a new ware useful in chemical work.

The Denver Fire Clay Company had a very fine display of highly refractory fire-clay crucibles of all sizes and a good line of cupels and scorifiers.

Chemical Porcelain.

Chemical porcelain was exhibited by The Coors Porcelain Company, Ohio Pottery Company and The Guernsey Earthenware Company. Each had a very complete display of porcelain crucibles, filter plates, casseroles, combustion capsules, evaporating dishes, concentric rings, filter rings, dye pots, funnels, desiccator plates, and many other forms used in chemical laboratories. The Coors Company showed a novel Bunsen burner made of glazed porcelain. Large evaporating dishes, both shallow and deep, in diameters up to twenty inches, showed successful advances along this line. The standards of workmanship and quality of ware were apparent advances made in porcelain manufacture.

Optical Glass.

There were two exhibits of optical glass, one by the Bausch and Lomb Optical Glass Company and the other by the Macbeth-Evans Company. Great advances have been made, due to re-

strictions in trade during the war. Optical glass has not only been made in the United States and from our own raw materials but of an excellent quality. The Bausch and Lomb Company displayed samples of their glass known as light flint No. 2, dense flint, extra dense flint, and boro-silicate crown, together with improved types of colorimeters, and refractometers. The Macbeth-Evans Company featured a large lighthouse lens made of their optical glass, together with a small exhibit of their well-known glassware.

Chemical Glassware.

The chemical glassware industry showed a great advancement in the quality of their products and in the manufacture of various shapes. Many manufacturers have brought their product up to the United States Bureau of Standards quality during the past few years. The exhibits have conclusively proven that the lack of mechanical skill in the making of various shapes of blown glassware is rapidly decreasing. Pieces of blown glassware, in the most intricate and accurately made forms, were a miracle to the ceramist in the way of shaping and forming the ware.

The Whitall-Tatum Company booth had a very effective display of their well and favorably known "non-sol" chemical glassware. The feature of the display was a 250 cc. "non-sol" beaker from which 220,000 cc. of distilled water had been driven off by boiling during a total of 1450 working hours.

The glass exhibit of the Corning Glass Works was very extensive, including pyrex glass for laboratory apparatus and baking dishes, chemical glass and laboratory apparatus. One of the features of the exhibit was a piece of square tubing. Colored glass tubing for various purposes formed another interesting portion of the display to the observer.

Booths of dealers in chemical glassware and porcelain were held by The Central Scientific Company, The Hanovia Chemical and Manufacturing Company, Eimer and Amend, Scientific Materials Company, E. H. Sargent and Company and Schaar and Company. The display of Eimer and Amend was of great interest as their work marked some new designs in glassware, together with

many pieces of delicately blown and ground ware. Many pieces of glassware were found to have been standardized according to shapes and sizes, bringing into this field a long-felt want, both to the user and to the manufacturer. Quartz-glass products, as shown by the Hanovia Chemical and Manufacturing Company, indicated advancements in workmanship as well as in increased size of ware. A glass blower, with the Central Scientific Company exhibit, attracted a great deal of attention as he shaped bulbs, pipettes, and other pieces of glassware.

The Thermal Syndicate Company display showed advances in "vitreosil" products for both the plant and the laboratory. As "vitreosil" is unaffected by the usual mineral acids or by sudden or extreme changes in temperature, its use has been extended to the manufacture of small chemical ware such as crucibles and evaporating dishes. Tubes of "vitreosil" ranging from $^{7}/_{16}''$ to $4^{1}/_{2}''$ diameter and 10 feet long were a feature of the exhibit.

Heat-Insulating Materials.

Very interesting exhibits were shown by the Celite Company and the Armstrong Cork Company on heat insulation. The Celite Company presented their product "Sil-O-Cel," in powdered and block form, one of the new features being a block three feet long which will broaden the field of use for this material. The Armstrong Cork Company featured their well-known "Nonpareil" brick. Both exhibitors had small models of kilns and furnaces, showing the application of their product and they also had electric furnaces in operation which demonstrated the superiority of their products over standard fire brick as heat-insulating materials.

ORIGINAL PAPERS AND DISCUSSIONS

THE CAUSE AND CONTROL OF "BLISTERING" IN SHEET-STEEL ENAMELING.

By Chester Treischel.

Introduction.

Every enameler is familiar with the defect known as "blistering," either the type evidenced by a separation of the enamel from the iron or with that one evidenced by a splitting apart of the steel sheet. The present investigation deals only with the latter of the two types. Blisters of this sort occur as a rule in "nests" which are merely aggregations of small blisters. They also seem to occur in parallel rows or striations, probably in the direction of rolling of the sheet, these points being clearly shown in the photographs. This form of blistering, because of its inherent peculiarity of manifesting itself when conditions in the plant are normal, has been the source of much worry to the men in charge, and it is toward determining the cause of and effecting a control over this defect, that this research has been directed.

It is well known that steel and iron absorb hydrogen with great avidity and that in so doing the properties of the metals are markedly altered. When iron is pickeled in acid, either hydrochloric or sulphuric, hydrogen is of course evolved. Enormous quantities of the gas are absorbed by the metal, but what is less well known is that only a part of the hydrogen can be expelled by heating the metal to 1000° C. With the aid of a vacuum it is possible to partially remove the gas at atmospheric temperatures. This fact has been demonstrated by Mr. S. P. Sweetzer, of the General Electric Co. Research Laboratory.

For sometime past it has been known that hydrogen can be

made to pass through comparatively thick walls of iron. By having a hollow vessel as cathode in an electrolytic cell, Dr. I. Langmuir succeeded in collecting the hydrogen which passed through the pores and was able to ignite it. It is said that a French investigator has succeeded in generating 40 atmospheres of pressure inside such a vessel under almost similar conditions. Another investigator, Mr. Stanley Meikle, has passed hydrogen through a steel diaphragm from one vessel to another. In Mr. Meikle's experiments he closed one end of a glass tube with a steel plate of approximately 30 mm. thickness. Inside of the vessel so formed he placed a neutral solution. The vessel was then immersed in a solution of hydrochloric acid for some time and upon testing the neutral solution it was found that it gave an acid reaction. There is, therefore, no lack of evidence that hydrogen will readily pass through a comparatively thick wall of steel or iron.

Perhaps no better illustration of the affinity of hydrogen for iron could be presented than the result of giving a spring a coating of japan. In certain japanning processes the iron or steel article is made the anode, the japanning being done electrolytically, which means that the hydrogen is being driven in the opposite direction and away from the iron; still the metal shows the same defect as is shown by all pieces of steel which have absorbed hydrogen, *viz.*, the piece becomes brittle, losing its properties of malleability and ductility.

The discrepancy of theory shown above is hard to explain. It must be that at the moment the hydrogen is generated at the anode and before it can escape into the stream of moving ions, it is absorbed by the iron, and this in spite of the electromotive force acting against it.

In the ordinary enameling process the metal parts are first "scaled," as the process of burning off the grease and forming a coating of iron oxide is called. The iron oxide is then removed by placing the parts in a warm bath of hydrochloric or sulphuric acid. The usual strength of this solution is in the neighborhood of 10 per cent acid, fresh acid being added from day to day to replenish the amount exhausted in combining with the iron and iron oxide.

In the manufacture of sheet steel for enameling, the steel is rolled to the desired thickness by passing the sheet between heavy rolls which are spaced at various distances from each other according to the thickness of sheet desired. In forming the ingot from which the sheet is rolled, air bubbles are incorporated unless the workmen are particularly careful in the pouring. In the rolling-out process these blebs are made much thinner in section but their size in a lateral direction is enormously increased so that an air bubble, which in the ingot was about the size of a pin head, would appear in the rolled sheet as a space of infinite thinness,-but perhaps of one or two square inches in area.

Noting these facts in connection with the manufacture of enameling-steel and also keeping in mind what has been said before with respect to the action of hydrogen upon steel, it is very easy for one to theoretically, at least, see whereby hydrogen from the pickling process might enter the iron sheets, the hydrogen ions congregating in the small interstices caused by the rolled-out air bubble and upon heating expand, causing a separation of the iron at this point as evidenced by the disagreeable blistering.

Experimental.

In order to investigate the probability of such an occurrence and in order to find out if this were really the true cause of this sort of blistering, the following experimental work was carried out on a commercial scale in our enameling plant:

Lot. No. 1.—Ten reflectors were picked at random from a batch of stampings, sand-blasted and enameled in the usual way.

Lot No. 2.—Ten pieces were taken from the same lot, scaled, pickled and enameled in the usual way.

Lot No. 3.—Ten pieces were dipped in a 10-per cent HCl solution and scaled in the usual way.

Lot No. 4.—Ten pieces were scaled, pickled in a 3-per cent HCl solution and enameled in the usual way.

Lot No. 5.—Ten pieces were scaled, pickled in 10-per cent. H_2SO_4 solution and enameled in the usual way.

Lot No. 6.—Ten pieces were dipped in 10-per cent H_2SO_4 solution and scaled.

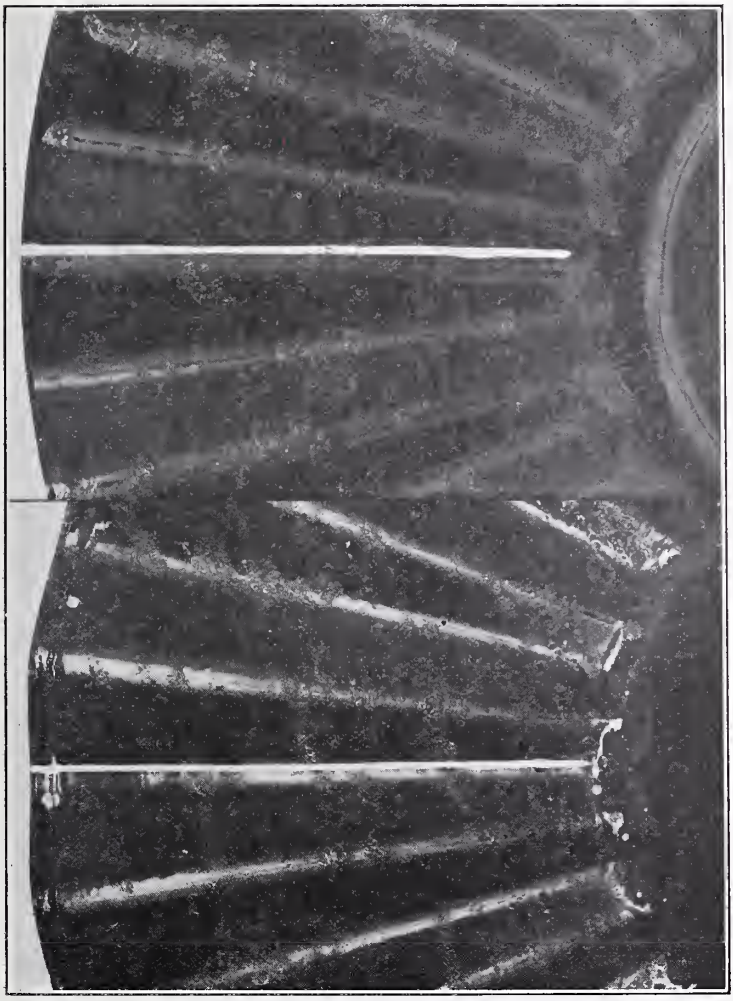


FIG. 1.—Enameled reflectors showing blow-hole defects which blistered when enameled.

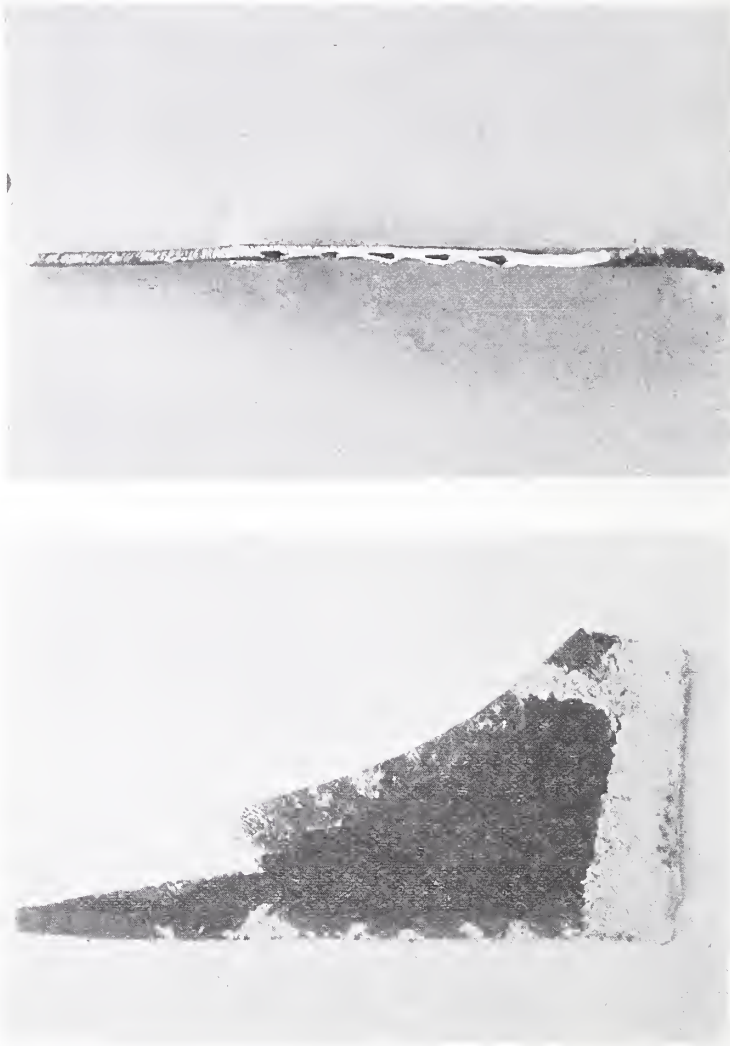


FIG. 2.—Sections enameled reflectors showing blow-hole defects which blistered when enameled.

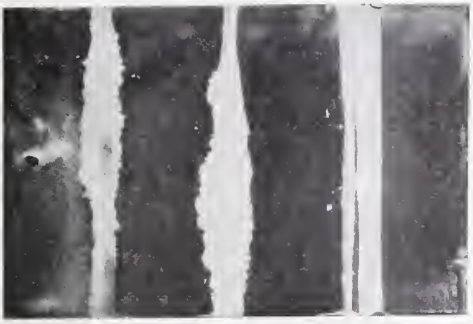


FIG. 3.—Pickled in acid.

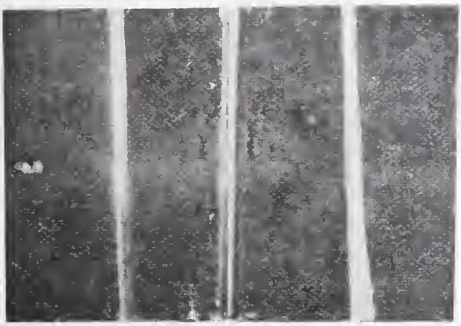


FIG. 4.—Vacuum treated.

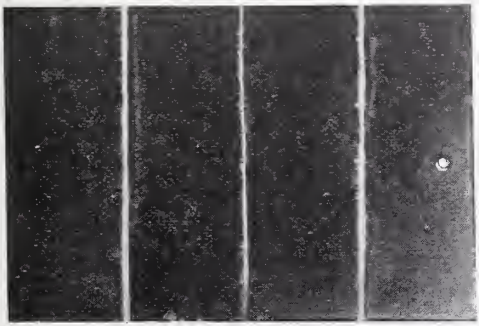


FIG. 5.—Sand blasted.

Lot No. 7.—Ten pieces were scaled, pickled in 3-per cent H_2SO_4 and enameled in the usual way.

Results.

The results of these experiments were as follows:

Lot No. 1.—None of these reflectors showed any signs of blistering.

Lot No. 2.—Every reflector in this lot showed marked blistering, the blisters varying in size from $^1/_2''$ diameter to blisters having an area of 2 or 3 sq. in.

Lot No. 3.—All of these reflectors showed blisters when taken from the furnace after scaling.

Lot No. 4.—None of these reflectors showed blisters.

Lot No. 5.—All the reflectors in this lot showed blisters similar in size and character to those shown in Lot No. 2.

Lot No. 6.—All of these reflectors showed blisters similar in size and character to those of Lot No. 3.

Lot No. 7.—None of these reflectors showed blistering.

Conclusions.

From the results of the above experiments it can be readily seen that the hydrogen certainly has much to do with the blistering in sheet-steel enameling. Those reflectors which were pickled in a strong solution of acid all showed blistering. This is no doubt due to the high concentration of liberated hydrogen ions which must necessarily take place when the iron is placed in an acid solution of such a strength. The reflectors which were pickled in a solution weak in acid necessarily were not acted upon as rapidly by the acid as in the case of the strong acid concentration. The concentration of liberated ions could not pile up so rapidly and therefore there was less penetration of the ions into the steel sheets. In the case of the lots which were dipped in acid before scaling, we have the evolution of hydrogen in the acid bath, together with the accompanying absorption of the hydrogen ions. These ions, lodging in the minute cavities caused by the rolled-out blow holes in the steel sheet, expand upon heating, causing the separation of the sheet accompanied by a noticeable blister at this point.

In order to make commercial use of the results of these experiments, it would be necessary to change the strength of the pickling solution to about 3 per cent. The time of pickling would not be very noticeably increased, although it would be necessary to have a workman rub each piece with a brush before washing—to help remove some of the adhering scale. In our case the quality of ware produced by such a change has resulted in practically no cases of blistering over a period of ten months, whereas with the use of the old method losses of 40 per cent were not uncommon on some lots of stampings.

General Electric Co.,
Schenectady, N. Y.

A METHOD FOR DETERMINATION OF THE VOLATILE MATTER IN OXIDES OF LEAD.

By Olaf Andersen.

For calculation of glass batch compositions it is necessary to know exactly the amount of PbO in the litharge or red lead used. If special tests show that the material contains no appreciable amount of metallic impurities, the obvious method is to drive off all volatile matter (CO_2, H_2O, and O present in PbO_2) by ignition. The residue may then be figured as pure PbO. In trying to do this, however, one will meet a difficulty at the very outset. The volatile constituents are driven off only slowly and to insure their expulsion in a reasonable period of time the oxide should be carried to its melting point. If, however, the sample is heated until melting takes place the containing crucible will be badly attacked. In order to overcome this difficulty the modification of the ignition method described below was worked out.

The ignition is done in a platinum crucible of almost any size or shape, but preferably of small diameter which will give the melt a relatively small surface and thereby diminish the loss by volatilization of lead oxide. To prevent any attack of the platinum the lead oxide sample is mixed with silica (sand or ground quartz) in the proportion of about 3 weights of PbO to 1 weight of SiO_2. On account of the formation of a low melting compound and eutectics in such a mixture the melting will take place at a temperature nearly 100° C, below the melting point of pure PbO (about 880°), and the result will be a silicate melt which does not appreciably attack the platinum.

The procedure for the determination is as follows: The sample is weighed in a platinum crucible. An amount of silica[1] equal to about one-third[2] of the assumed amount of PbO in the

[1] The silica used here must not merely be dried at 100° or 200° but, previous to use in this method, it should be ignited to 1000° to completely remove volatile matter. This procedure is advisable in all cases and absolutely necessary with silica derived from carbonate-carrying sands.

[2] At the Charleroi plant we used 4 of lead oxide to 1 of silica.

sample is added to the latter and the two are carefully mixed in the crucible; finally, a thin layer of silica is spread over the top of the mixture. The crucible with full charge is weighed and placed inside an electric furnace previously heated to 800°. The temperature must be accurately measured and must not be permitted to change considerably. The heating at 800° is continued for about 20 minutes and the temperature is raised as quickly as possible to about 1000° where it is held for about 15 minutes more. Then the crucible is taken out, covered to prevent the cracking glass from flying out of the crucible, cooled and weighed. The loss in weight is the amount of volatile constituents present in the lead oxide and, in case there are no other impurities, the remainder of the original sample is pure lead oxide, PbO.

The method has been used to a considerable extent in connection with our optical glass work at the laboratories of the Pittsburgh Plate Glass Company at Charleroi, Pennsylvania, and the Spencer Lens Company at Hamburg, New York. The results checked well with a few direct analyses made on some of the samples. The average run of the figures obtained on litharge is indicated by the following examples:[1]

Average of 14 samples litharge	98.99 per cent PbO
Maximum of 14 samples litharge	99.55 per cent PbO
Minimum of 14 samples litharge	98.00 per cent PbO
Top layer of barrel which had stood open 4–6 weeks	97.27 per cent PbO
Top layer of barrel which had stood open 4–6 weeks	96.61 per cent PbO

GEOPHYSICAL LABORATORY,
CARNEGIE INSTITUTION OF WASHINGTON,
August, 1919.

[1] Data furnished by R. H. Lombard.

THE VOLATILIZATION OF LEAD OXIDE FROM LEAD SILICATE MELTS.

By Olaf Andersen.

The experiments described below were undertaken in the course of the Geophysical Laboratory's work on optical glass during the war, in order to determine the order of magnitude of the loss due to volatilization of lead oxide from lead silicate melts under certain conditions. An exact determination of such data, with perfect control of all determining factors, would involve a very comprehensive set of experiments, but approximate results may be obtained with simple means.

The material for the experiments consisted of homogeneous glasses containing, apart from incidental impurities, only lead oxide and silica in various proportions. The heatings were done in the same electric furnace in which the experimental glasses had been made, with the opening covered but not tightly closed. The broken glass was filled into a platinum crucible, which had been previously tested for approximate constancy of weight under the conditions of the subsequent experiment. The crucible with charge was heated until the glass formed a melt with level surface, then cooled and weighed. The furnace was then regulated to the temperature desired for the experiment and the crucible was introduced and held for a certain time, taken out, cooled, and weighed again. The difference between the two weighings gives the loss in the melt and as the only volatile constituent in every case was lead oxide, the total loss may be safely calculated as PbO. The area of the surface was then measured.

A few tests showed that the actual loss from the same melt at the same temperature was proportional to the area of the exposed surface and independent of the weight of the melt. The factor determined was therefore the rate of loss per unit of the exposed surface of the melt. The units used were 1 hour, 1 square centimeter, and 1 milligram.

It was found that the rate of loss decreased with a prolonged heating when the melt was left without stirring, but remained constant when stirring was applied. The explanation of this is that diffusion alone works very slowly and will only partly offset the change in composition due to volatilization of lead oxide from a thin film next to the surface of the melt. This film will therefore become gradually richer in silica, at the same time growing lighter than the underlying melt so that it will remain at the top as a protecting film and thereby diminish the rate of volatilization. If, on the other hand, the melt is stirred the rate of volatilization will remain practically constant for a long time because the total loss of lead oxide will be distributed throughout the melt and the actual change in composition of the whole melt, and consequently also of the surface film, will be small. To obtain the exact rate of loss for a certain melt it should therefore be necessary to stir it during the entire period of heating.

This, however, would involve a rather complicated arrangement of experiments and would consume much time. Tests were therefore made to find out whether the rate of loss computed from a short heating without stirring would not be materially the same as the one obtained through a prolonged heating with stirring. The experiments in Table 1 were carried out with a melt of the composition: 70 per cent (by weight) PbO, 30 per cent SiO_2, at a temperature of 1300°.

TABLE 1.

Volatilization of PbO under Different Conditions.

Time of heating.	Condition.	Rate of loss mg./hr./sq. cm.
20 hours	No stirring	8
15 min.	No stirring	23
1 hour	With stirring	22

These tests show that the rate obtained on the basis of a short heating without stirring is practically identical with the one obtained by a somewhat longer heating with continual stirring.

It is also seen that a prolonged heating without stirring gives a much lower rate of loss than either of the other methods.

In view of these facts the computed rates of loss were based on a short heating without stirring of the melt. A period of 15 minutes was found convenient.

The results are tabulated in Table 2 and plotted in Fig. 1.

TABLE 2.

Volatilization of PbO from Pure Lead Silicate Glasses.
(Loss in mg./hr./sq. cm.)

Percentage of PbO in glass.....	85	78.7	75	70	65	60
Temperature °C.						
1400	318	131	91	60	46	33
1300	100	40	30	23	16	13
1200	37	11	8	7	5	4
1100	13	4	2	2	1	1
1000	8	2	1	1	..	..
900	1	1	..	..	..	..

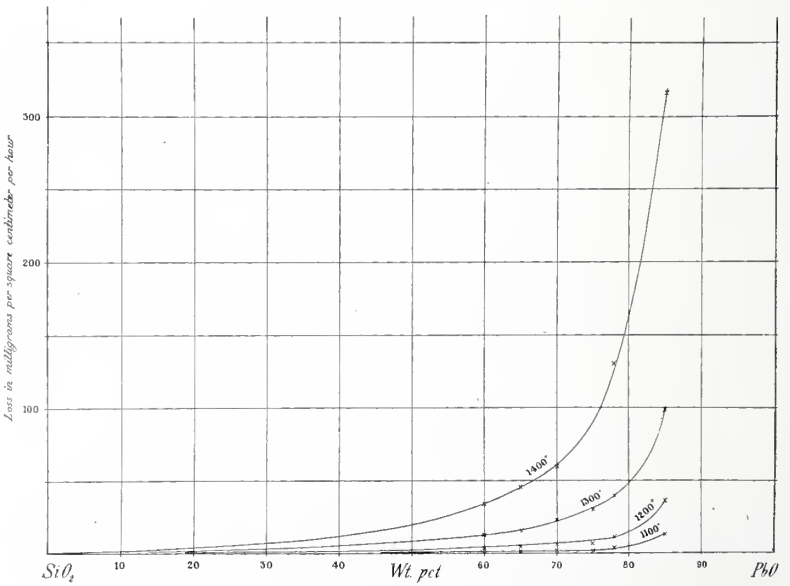


Fig. 1.

In several cases the refractive indices at various depths of the melt were determined after certain heatings. It was invariably found that a thin layer near the surface, and sometimes a film creeping up along the inside of the crucible, showed a considerably lower refractive index than the underlying glass. Table 3 contains a few examples of this relation.

TABLE 3.

Volatilization of PbO from Surface of Glass Melt.

Time of heating.	Composition.		Refractive index after heating.		Remarks.
	SiO_2.	PbO.	Middle of glass.	Surface film.	
2 hours	21.3	78.7	1.888	1.860	
3 hours	11.9	88.1	2.09	1.63	Surface film creeping up inside of crucible
2 hours	35.0	65.0	1.724	1.70	

During the experiments it was often noticed that small isolated patches of thin glass film on the sides of the crucible very soon lost so much lead oxide that silica began to crystallize out, the glass becoming white. Under the microscope the crystals were found to be tridymite, which, according to Fenner,[1] is the stable form of silica between 870° and 1470°.

To apply the results to actual glass-melting one may take the following example: A glass containing 60 PbO and 40 SiO_2 is heated for 48 hours at 1300°. Suppose the area of the surface to be 7500 sq. cm. (approximately the area exposed in a pot of 39 inches inside diameter). The loss according to the diagram (Fig. 1) is 48 × 7500 × 13 mg. or 4.68 kg. On a 1000 kg. charge this means a loss of about 0.5 per cent of the weight of the glass, or 0.8 per cent of the PbO. This will have an influence on the refractive index of about 2 in the third decimal place. Similarly, if this glass were fined at 1400°—the usual practice—the loss[2] by

[1] C. N. Fenner, *Am. J. Sci.*, **36**, 331–384 (1913); *J. Soc. Glass Tech.*, **3**, 116–125 (1919).

[2] With the shortened melting and fining schedules used more recently the losses would be approximately half those calculated.

volatility would be 12.2 kg. PbO which is equivalent to 1.22 per cent on the weight of the glass or 2.0 per cent on the weight of PbO. Since melts of a particular kind of glass are always given as nearly as possible the same kind of treatment, the loss will be subject to only small changes from melt to melt and any considerable variation in refractive index is probably not to be ascribed to a variation in the amount of lead oxide lost through volatilization from the surface of the melt. If, however, the heating schedule of the same melt has been considerably changed a noticeable variation in optical properties due to change in loss by volatilization must be expected in all melts rich in lead oxide. This factor is probably largely responsible for the considerable variations in optical properties found in melts, presumably of the same composition, made experimentally on a small scale to determine the relation of optical properties to composition in the lead glasses.[1]

That volatilization is a factor of large magnitude is apparent to anyone who has observed the dense "smoke" issuing from the opening of a covered pot containing a lead glass melt which, on account of accident, has been taken from the furnace at the fining temperature. The vapors in this case consist not only of lead oxide but also of alkalies and boric oxide; the opacity and apparent magnitude of the cloud is increased by the condensation of these vapors to solid particles on striking the cold air, but even after allowing for this impression the amount of material evolved is surprisingly large.

The losses of PbO observed in large-scale operations have at times actually reached figures in excess of 5 per cent of the PbO content. These large losses were partly caused by volatilization as above described, but also, in addition, by litharge being mechanically carried out of the pot by gases such as O, CO_2, and oxides of nitrogen derived from the decomposition of batch materials. Such losses were particularly noticeable with the extremely finely-divided sublimed litharge used so extensively in the last year of the war. The rate of filling in the batch would be an important factor in determining such losses, and likewise

[1] Data to be presented in a subsequent paper.

also the practice in regard to stoppering the opening of covered pots, when such pots were used.

Summary.

Experiments were made on the amount of PbO volatilized from the surface of lead silicate glasses at temperatures from 900° to 1400° C. It was found that the volatilization from an unstirred glass in 15 minutes took place at practically the same rate as from a stirred glass heated for a longer period. The rate of volatilization falls off considerably during a long heating if the glass is not stirred, as a film considerably lower in PbO forms on the surface of the melt, into which PbO must diffuse from below before it can escape. The rate of volatilization of PbO at temperatures usually used in optical glass manufacture would seem from these experiments to be small enough so that variations in refractive index due to volatilization are not to be expected if the procedure is reasonably constant from melt to melt, but large enough to cause considerable variations in index if the melting schedule is changed.

Geophysical Laboratory,
Carnegie Institution of Washington,
August, 1919.

DISCUSSION OF AN ARTICLE BY ELLIS LOVEJOY, ENTITLED, "NOTES ON FIRE CLAYS OF THE NORTHERN APPALACHIAN COAL BASIN," IN THE MAY NUMBER OF THE JOURNAL OF THE AMERICAN CERAMIC SOCIETY.[1]

By George H. Ashley.[2]

First. The stratigraphic position of the flint clays as described by Mr. Lovejoy has been questioned, but I think that Mr. Lovejoy is correct in assigning the principal deposits in the northern Appalachians to the horizons of the Sciotoville, Upper Mercer, Lower Kittanning, and Upper Freeport. These are by no means all of the horizons but flint clay is widespread at at least the last three of those horizons. At Bolivar, Pa., and some other places, the flint clay is below the Upper Freeport limestone and in a measure distinct from the under-clay of the Upper Freeport coal, but at hundreds of other places the flint clay is so close below the coal that for all practical purposes the horizon may be spoken of as at the horizon of the Upper Freeport clay. Flint clay has been found at a few localities under what has been correlated as the Upper Kittanning coal. I do not recall a single instance of its occurrence under the Middle Kittanning coal though it may so occur. Its occurrence under the Lower Kittanning coal, that is, between that coal and the Vanport limestone, is so common as to form one of the "earmarks" of that coal. In this position it is frequently referred to as the Clarion flint clay, as it occurs at this horizon in the town of Clarion and at scores of places in every direction from the town. There seems to be little doubt that the upper clay, all through the Woodland district of Clearfield County, Pa., is under the Lower Kittanning coal or else the Allegheny stratigraphy of central Pennsylvania must be recast. It is found at the same horizon from central Pennsylvania as far southwest as northeastern Kentucky. In

[1] *J. Am. Ceram. Soc.*, 2, 374 (1919).

[2] By permission of the Director, U. S. Geological Survey.

scores of places its position between the Vanport limestone and the Lower Kittanning coal is so clear as to leave no doubt as to its horizon. Recent intensive work in the Mount Savage area of Maryland, by Messrs. Harvey Bassler and Charles K. Swartz of the Maryland State Survey, extended by studies into the type areas of Pennsylvania (Kittanning, Brookville, Mercer, and others), and recent work by the Federal Survey in Somerset County, and stratigraphic studies in the Beaver River and Allegheny valleys, have indicated the possibility that some of the so-called Mercer flint clays may belong at the base of the Allegheny or in the horizon of the Clarion and Brookville coals. At the town of Clearfield, Pa., and at a few other places, there is a remarkable succession of flint clays extending from just below the Lower Kittanning coal downward for 60 feet or more, interbedded with a number of thin coals.

Second. I am inclined to believe with Mr. Lovejoy that flint clay may be the result of secondary chemical changes, yet the persistent recurrence at a few definite horizons argues for there having been original differences in the deposits in which they occur from the other argillaceous deposits of the region. Thus, it is a common experience, particularly with the clay at the Lower Kittanning horizon, to find the flint clay confined to the front of the hill, where it is under light cover, and that as the bed is followed under the hill the flint clay disappears. This suggests secondary action by surface waters. Again, the extreme irregularity of the bodies of flint clays in the general mass of soft clay, as figured by me in the report of the Topographic and Geologic Survey Commission of Pennsylvania, 1906–8, page 316, suggests secondary action. On the other hand, where the bed of flint clay can be traced almost continuously for many miles it would appear to be a primary deposit, or at least based on differences in the original argillaceous deposit, in contrast with other adjacent argillaceous deposits. For example, there are in Kentucky two horizons of this kind. In northeastern Kentucky there is quite an area where the thick clay under the Lower Kittanning coal has in the middle a layer of flint clay only a few inches thick. Again, in southeastern Kentucky there is a coal bed known as the Fire Clay coal, the Hayden, or No. 4 bed, which at hundreds

of openings shows a parting of flint clay. This has been traced over a large area through Letcher, Leslie, Harlan, Clay, Knox, Bell, and Whitley counties, Kentucky, and into Claiborne, Campbell, Anderson, and Scott counties, Tennessee, forming the best key horizon in the whole region. Again, in north Beaver County and south Lawrence County, Pennsylvania, and apparently at the same horizon elsewhere in Pennsylvania, there is a deposit of flint clay entirely unassociated with either soft clay or coal. It occurs in the shale of the Mahoning interval above the Upper Freeport coal. It is not commonly a distinctly marked horizon but a narrow belt in which the shale gradually changes over into a flinty shale and locally into a ferruginous flint clay, and at the top changes back into the overlying shale. The shale where affected breaks down like crushed flint, crunching under buggy wheels like broken glass. This belt in the area mentioned is so regular as to serve as a key horizon in mapping. These occurrences at widespread horizons seem to indicate that flint clays if of secondary origin originate only in deposits having characteristic original differences from other shaly or clayey deposits. What those differences were or how they may have originated, I can not say.

Third. Mr. Lovejoy's distinctions of the flint clays at the four principal horizons agrees in general with my observations though I have not found it safe to put much weight on those apparent differences for stratigraphic purposes.

Fourth. In so far as Mr. Lovejoy has put weight on Stout's theory of origin of clays associated with coal, I fear he is on the wrong track. This is not the place to discuss that theory, but I believe the examination of several thousand under-clay sections will convince any one that the under-clays are not uniform vertically, as demanded by the theory, but are generally, if not universally, different at the top and bottom, the bottom usually being more sandy if the underlying bed is a sandstone, and in many, if not most places, grading into the underlying bed, whatever that may be. Nor can any one trace clay partings in coal beds from mine to mine, particularly in horizons where the benches spread apart and reunite irregularly, and watch closely the change in the character of the partings from point to point

without becoming convinced that he is dealing with an ordinary widespread sedimentary deposit. Some of these partings are remarkably persistent and uniform over large areas, but is not the very delicate adjustment of physical conditions necessitated by Stout's theory much more difficult of imagination than the common one of a widespread flooding by mud-carrying waters? Certainly the shoreward edges of such partings, which generally tend to thicken and become sandy, indicate water deposits. The theory would have difficulty in accounting for sandstone partings, which in some areas have a wide enough extent to serve as keys in the correlation of the beds. That underclays *may* have been affected by the action of plant growth going on over them is clearly evidenced by the apparent effect of such growth on sandstones, which in many places underlie the coals in place of clays. Such sandstones, especially where penetrated by Stigmaria, are commonly whitened next to the coal, locally to depths of 4 to 5 feet or more, and this white sandstone is found to be freer of alkalies than the adjoining sandstones, and is locally called ganister and so used in the lining of furnaces.

Topographic and Geologic Survey of Pennsylvania,
Harrisburg, Pa.

GLASS-SAND RESOURCES OF VIRGINIA.

By Thomas L. Watson.

Introduction.

The resources of Virginia in quartz sand or its hard rock equivalents suitable for glass-making are very great. Quartz in the form of sand or hard rock desirable for purposes of glass manufacture occurs in each of the three major physiographic divisions of the State, and is produced at present in each province for such use. In many localities the sandstones and quartzites are of exceptional purity. Many of them are favorably located for quarrying and to lines of transportation, and are near to abundant supplies of high-grade coal.

There are four plants in Virginia engaged at present in the manufacture of glass, located at Alexandria, Richmond, Lynchburg, and Salem, with a fifth one expected to begin operating shortly at Norfolk. Of the four producing plants two derive their supply of sand from Virginia while the others obtain their supply from West Virginia. The total production of glass-sand, which is not very large, is consumed within the State.

Kinds of Material.

The kinds of silica found in Virginia in quantity and of sufficient purity (see table of analyses on page 797) to be utilized in making glass include (1) quartz sand, (2) sandstone and quartzite, and (3) vein quartz including some pegmatite occurrences. These materials probably have not equal value for glass-making and, under present conditions, some of them may not be usable, although well located and occurring in quantity and of sufficient purity. The distribution and character, including analyses of these materials, are briefly summarized below by physiographic provinces.

Coastal Plain Province.

The Coastal Plain or Tidewater Virginia, the easternmost province, comprises about one-fourth the total area of the State.

The major streams which cross it are navigable for the entire width of the province and with the lines of railway afford excellent transportation facilities.

The Coastal Plain province is composed of loose or locally indurated sediments, of which sands of different kinds form an important part. The sands vary greatly both as to size of grain and purity. In many localities they consist of pure quartz grains but more often they are admixed with more or less clay and iron oxide, and may carry small grains of other minerals than quartz, such as magnetite, ilmenite, etc., but not in harmful amounts in some of the deposits. Glauconite, an iron-bearing mineral, is usually an abundant constituent in many of the Eocene sands which are of little or no value for glass manufacture when this mineral occurs in appreciable quantity.

The better grades of the Coastal Plain sands have been employed in a variety of uses but in greatest quantity for building purposes. Some of the sands are pure enough to be utilized for glass-making, but thus far they have been so employed only to a very limited extent.

Extending from Cape Henry southward into North Carolina, sands dune are prominently developed along the coast, forming a conspicuous feature of the coastal topography. The material is loose beach-sand accumulated landward by the wind. Small shipments of this sand in the vicinity of Cape Henry are made at present to one of the Virginia plants for glass-making. Because of their extent and favorable location (only a few miles east from the city of Norfolk), these sands are worthy of careful investigation in order to determine their desirability for glass manufacture.

Extending along the western margin of the Coastal Plain, from Fredericksburg northeastward to the Virginia boundary, is a belt of Cretaceous sands sufficiently indurated locally as to have been formerly quarried for building stone. They are essentially light-colored sands, composed in places almost entirely of pure variable-size quartz grains, but they frequently contain much kaolinized feldspar. It seems probable that, in certain localities within this belt, the sands might be utilized to advantage in the manufacture

of at least the lower grades of glass. So far as we are aware, however, no attempt has yet been made to use them.

Piedmont Plateau Province.

As defined at present, the Piedmont Plateau province comprises that part of Virginia included within the fall belt on the east and the southeast slope of the Blue Ridge Mountains on the west. It is composed dominantly of a complex of the older crystalline silicious rocks, although several areas of Triassic rocks are comprised within its limits. Belts of metamorphosed sediments yielding crystalline limestones, quartzites, and slates of early Paleozoic age also occur.

Within the Piedmont province are distributed several kinds of highly quartzose materials, including quartzite, quartz veins, and pegmatite bodies, some of which are sufficiently pure and otherwise desirable for glass manufacture.

The stream sands deposited along river courses in the Piedmont province are usually too impure to be seriously considered for purposes of glass manufacture. Most of the quartzites are likewise too impure to be used in glass-making, since they may carry large but varying amounts of either micaceous minerals, feldspar, epidote, or iron oxides (chiefly magnetite and ilmenite, and in places hematite). In some localities, however, the quartzites are entirely white in color; are composed almost wholly of pure quartz, with inappreciable amounts of other minerals; and could be used to advantage in glass-making.

Only one locality in the Piedmont Plateau is yielding quartzite or its disintegration product, quartz sand, at present for the manufacture of glass. A white quartzite of Lower Cambrian age, located near Stapleton in Amherst County, about 12 miles north of east from the city of Lynchburg, is being utilized by the Lynchburg plant for the making of glass. A partial analysis of a sample of this quartzite is given in the table of analyses on page 797. The sample yielding the analysis, however, contains, besides quartz, considerable white, silvery mica (sericite). Other analyses of samples of the quartzite indicate more than 95 per cent of silica, with only a trace of iron. Fusion tests made on a sample of the rock yielded a very satisfactory grade of glass. Similar quartzites

TABLE 1.—ANALYSES OF VIRGINIA GLASS SANDS.[1]

Piedmont Plateau Province.

	1.	2.	3.	4.	5.
SiO_2	91.403	94.37	99.870	99.638	99.33
Fe_2O_3	0.476	0.07	0.930	0.025	....
CaO	0.260	....	....	....	....
Ignition	1.780	0.93	0.068	0.184	....
Undetermined	6.081	4.63	....	....	0.67
	100.000	100.00	100.868	99.847	100.00

Mountain Province.

	6.	7.	8.	9.	10.	11.
SiO_2	99.66	99.08	99.471	98.78	96.99	98.546
Al_2O_3	0.18	0.18	....	0.27	0.01	....
Fe_2O_3	0.09	0.32	0.076	0.41	0.02	0.099
MgO	....	...	....	...	0.07	....
CaO	....	...	0.030	...	0.80	....
Na_2O }	0.07	0.40	....	...	1.60	....
K_2O }						
TiO_2	....	...	....	...	...	0.060
Ignition	....	...	0.129	...	0.31	0.327
	100.00	99.98	99.706	99.46	99.80	99.032

[1] Nos. 1 and 2. Cambrian quartzite near Stapleton, Amherst County, Virginia (S. D. Gooch, analyst).

No. 3. Silex (vein quartz), Madison Courthouse, Madison County, Virginia (S. D. Gooch, analyst).

No. 4. Silex (vein quartz), 1 mile east of Boyd Tavern, Fluvanna County, Virginia (S. D. Gooch, analyst).

No. 5. Silex (vein quartz), near Scottsville, Albemarle County, Virginia (J. B. Weems, analyst).

Nos. 6 and 7. Cambrian (Erwin) sandstone from Locher place (Glasgow Clay Products Company), near Glasgow, Rockbridge County, Virginia.

No. 8. Silurian (Clinch) sandstone near Kermit, Scott County, Virginia (S. D. Gooch, analyst).

No. 9. Silurian (Massanutten-Tuscarora) sandstone, Catawba Mountain, 9 miles north of Salem, Virginia. (By courtesy of the Salem Glass Company, Inc., Salem, Virginia.)

No. 10. Catawba Valley sand (Massanutten-Tuscarora sandstone), 9 miles north of Salem, Virginia (H. H. Hill, analyst).

No. 11. Cambrian (Erwin) sandstone, C. & O. railroad quarry, east of Basic, Augusta County, Virginia (S. D. Gooch, analyst).

of equal purity and of the same geologic age are found in other localities in the Piedmont province, but have not yet been drawn on as a source of raw material for glass manufacture.

Large bodies of exceptionally pure quartz are widely distributed over many parts of the Piedmont province which, judging from the analyses given on page 797, could be utilized in many cases to advantage in glass-making. The quartz of the numerous pegmatite bodies occurring in the Piedmont province is probably of less importance because of the size of the pegmatites and the irregular distribution of the quartz in them, in this connection than either the quartzites or the vein quartz, although the quartz can be readily separated from the feldspar, both of which are used in the manufacture of pottery.

Mountain Province.

This province embraces the western part of the State; its eastern boundary is the southeast slope of the Blue Ridge and its western limits mark the boundary between Virginia, West Virginia, and Kentucky. It includes the following topographic divisions: (1) The Appalachian Mountains to which the name Blue Ridge is applied in Virginia; (2) the Appalachian Valley which is divided lengthwise into an eastern part known as the Great Valley and a western part referred to as the Valley Ridges; and (3) the Appalachian Plateau, the Virginia portion of which includes the extreme southwestern part of the State adjacent to Kentucky and West Virginia. The Appalachian Mountains and Appalachian Valley provinces extend in a general southwest direction over 300 miles from the northern to the southern boundary of the State.

Of the subdivisions of the Mountain province the Appalachian Valley, which is composed of folded Paleozoic sedimentary rocks ranging from Cambrian to Carboniferous (Mississippian) in age, contains vast glass-sand resources in the form of sandstones and quartzites. Notwithstanding this fact there is only one glass-making plant operating at present in this part of the State.

The formations of the Appalachian Valley that either contain abundant supplies of silica suitable for glass-making or are worthy

of careful investigation for such use may be tabulated in the order of their age, beginning with the oldest, as follows:

Cambrian:

1. Unicoi sandstone which includes the Weverton sandstone and Loudoun formation in northern Virginia and adjacent parts of West Virginia.

2. Erwin quartzite which includes the Antietam sandstone of northern Virginia and adjacent parts of West Virginia and western Maryland.

Silurian:

3. Clinch sandstone which includes the Tuscarora (Massanutten) sandstone of west-central and northern Virginia, and adjacent parts of West Virginia.

4. "Rockwood" formation.

Devonian:

5. Oriskany ("Monterey") sandstone.

Mississippian:

6. Pocono sandstone which includes the Price sandstone of southwestern Virginia.

The sandstones of each of these formations have wide general distribution in the Appalachian province, but owing to their greater purity in many localities the sandstones of the Cambrian and Silurian systems must be considered the most important sources of silicious material for glass manufacture. As indicated from the analyses on page 797 and from the descriptions below, these sandstones are remarkably free from impurities in places, especially iron, which is essentially absent.

Cambrian Sandstones.—The principal development of the Cambrian sandstones in Virginia, including the Unicoi (Weverton and Loudoun of northern Virginia), and Erwin quartzite (Antietam of northern Virginia) is along the west foot of the Blue Ridge or at the border of the Appalachian Valley and the Appalachian Mountain (Blue Ridge) provinces. In their normal sequence the Unicoi and Erwin are separated by the Hampton (Harpers) shale, which formation is estimated to be at least 400 feet thick in northern Virginia and 600 to 800 feet in southwestern Virginia.

Because of their greater resistance to weathering the sandstones and quartzites of these formations form low knobs and high ridges of considerable prominence in places in front of the main Blue Ridge. "The Erwin quartzite is one of the most conspicuous formations in the region because it makes such prominent rocky ridges and because of its conspicuous white color."[1] Over much of the region the beds of these two formations, especially those of the Unicoi, are of no value as glass sands, but in many localities the massive white to gray beds of sandstone are of sufficient purity to be of importance as a source of sand for glass-making.

The Unicoi formation which directly overlies the crystalline rocks, includes sandstones, arkoses, conglomerates, and quartzites. "In the northern part of the region these beds range from soft arkose through harder arkosic sandstones to hard, gray sandstone and dark, ferruginous quartzite, and associated beds of slaty, argillaceous sandstone. Some of the basal beds have rounded grains and small pebbles of quartz which are usually clear and transparent, though some have an opaline-blue color. Besides quartz there are grains of feldspar, generally chalky white from weathering, and considerable clay and iron oxide."[2]

The Unicoi formation in northern Virginia is probably not less than 1,750 feet in thickness and is probably thicker in southwestern Virginia. Except in northern Virginia and possibly in some places in southwestern Virginia, the Unicoi sandstones are not generally of glass-making quality. East of the main Blue Ridge in Loudoun County some beds of the Unicoi (Weverton) are very white and pure, being composed almost entirely of quartz grains and apparently could be used to advantage in glass-making.

The Erwin quartzite is a massive white rock, the outcrops of which make prominent ridges throughout the region, and in northern and central-western Virginia, especially, it forms a potential source of unlimited supplies of glass sand. "The western foothills of the Blue Ridge over most of the region are composed of this rock, and it also caps many of the ridges of an inner row where there are several lines of ridges. In the northern part of

[1] Va. Geol. Surv., *Bull.* **17**, 17 (1919).

[2] *Ibid.*, **17**, 13 (1919).

the area the formation is generally composed of three massive cliff-making ledges separated by thinner bedded sandstones. . . . . "The cliff-making ledges consist largely of massive beds of dense, white quartzite, some of which are 15 to 20 feet thick without a visible trace of bedding."[1] Analyses of the Erwin quartzite are given in the table of analyses on page 797.

Over much of the southwest Virginia region, especially in Smyth and Wythe counties, white, hard, massive, vitreous quartzite is least abundant of the beds composing the Erwin, and hence the value of the Erwin sandstone over this part of the State is of less importance as a source of supply of glass sand than in the central-western and northern parts of Virginia.

Silurian Sandstones.—Of the two sandstones of Silurian age the Clinch and the coarse, white sandstone next below the Clinch, or the top member of the "Rockwood" formation (Clinton), the former is much the more important for glass-making.

The Clinch sandstone of southwestern Virginia and its equivalent Tuscarora (Massanutten) sandstone of west-central and northern Virginia, is a massive, coarse, white sandstone or quartzite that forms most of the more prominent valley ridges in southwest Virginia. It ranges up to 300 or 400 feet in thickness and is prominently developed in Clinch Mountain which is the most conspicuous of the Valley Ridges. Under favorable conditions the sandstone weathers in places to a white sand composed almost entirely of pure quartz grains.

The glass plant located at Salem, Virginia is using the equivalent of the Clinch sandstone (Tuscarora) which is derived from Catawba Mountain, about 9 miles from Salem, in the manufacture of glass. The rock is crushed and screened and is then ready for use. An analysis of this rock is given in the table of analyses on page 797.

At or near Kermit, a station on the Carolina, Clinchfield & Ohio railroad, Scott County, is one of the most important localities in the State from which a high-grade glass sand of excellent quality can be obtained for glass-making. Here the Clinch sandstone has good development in beds of exceptional purity. The

[1] Va. Geol. Surv., *Bull.* **17**, 15 (1919).

sandstone is composed almost entirely of moderately rounded, fairly uniform-size grains of quartz of great purity, the total ferric oxide amounting to only 0.014 per cent, as determined by the Pittsburgh Branch of the Bureau of Standards on a sample submitted by the writer. Fusion tests made with a second sample of the sand gave an excellent color, even without the use of a decolorizer. This quality of sand should be of considerable interest to manufacturers of optical glass and of other kinds of fine glass. (See table of analyses on page 797 for an analysis of the Clinch sandstone near Kermit.)

Devonian Sandstone.—The Oriskany ("Monterey") sandstone has wide distribution in western Virginia, chiefly in the Valley Ridges province where owing to the hardness of the rock its outcrops usually form knobs and ridges that are often prominent. The formation, which may range up to several hundred feet in thickness, is composed of a hard, fine- to medium-grained calcareous sandstone of light buff to bluish gray color. The quartz grains are cemented by lime carbonate and under favorable conditions the rock weathers readily to sand and loose fragments. The Oriskany sandstone has not been quarried in Virginia for glass-making, but in some localities in the State where the rock is essentially free from iron and other injurious impurities it seems worthy of investigation for such use. It is well known that this sandstone is quarried and used in Pennsylvania and West Virginia for glass-making and in the latter State it is the most important glass-sand horizon yet developed.

Under this heading are included sandstones, mostly unfossiliferous, which may be of Salina (Silurian) age, and which in one locality at least are of marked purity. In a section measured by Mr. G. W. Stose, along the Chesapeake and Ohio railroad on the east side of James River gap through Rathole Mountain, Eagle Rock, a 55-foot thickness was shown of a pure white, sugary, granular sandstone, of probable Salina age, which crumbles to white sand on weathering. The purity, thickness, and location of this sandstone immediately on the railroad render it especially worthy of careful investigation for probable use in the manufacture of the better grades of glass. Some of the sandstone beds below

this one in the section are also quite pure and of considerable thickness, and will probably prove to be of value for the same purpose. The Tuscarora sandstone of hard, white to gray color beds, and alternating with soft, white sandstone beds, poorly exposed, are shown in the same section. These sandstones (Salina? and Tuscarora) are worthy of careful examination and analysis to determine their desirability for glass-making.

Mississippian Sandstone.—The Pocono sandstone of west-central and northern Virginia and its equivalent, the Price sandstone of southwestern Virginia, is a hard, ridge-making rock that is subject to considerable variation in composition in different localities. It frequently carries thin beds of coal near the top and is apt to be conglomeratic in the lower or basal portion. Because of its iron content and the presence of other impurities, together with non-uniformity of texture as generally shown in most of its outcrops over southwest Virginia, the Price sandstone has no value for glass-making, but in west-central Virginia its equivalent, the Pocono, is developed as a heavy bedded white-to-buff quartzite of apparently sufficient purity as to encourage the belief that in some places at least it might be used.

UNIVERSITY OF VIRGINIA.

RELATION BETWEEN THE COMPOSITION AND THE THERMAL EXPANSIVITY OF PORCELAINS.[1]

By F. H. Riddle.

Considerable work has been done in the study of the thermal coefficients of expansion of porcelain bodies, the bulk of which has been contributed by the membership of this Society.[2]

Most of this work has been carried out with a view of determining the safe range of body composition with reference to the behavior of the glaze. In some cases the bodies were fired to a given temperature, irrespective of the maturing or vitrification point, so that, in fact, the bodies compared differed not only in composition but also in structure.

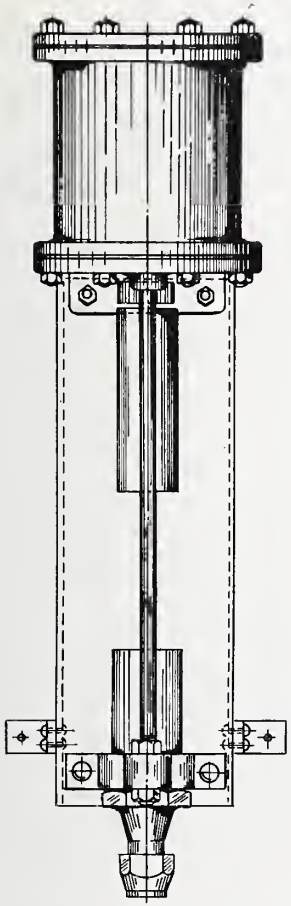
The present work was carried out in connection with a study of porcelains required for severe duty as regards temperature changes and electrical resistance. It was found necessary, therefore, to determine the thermal expansions of a number of compositions, all of which were fired to maturity.

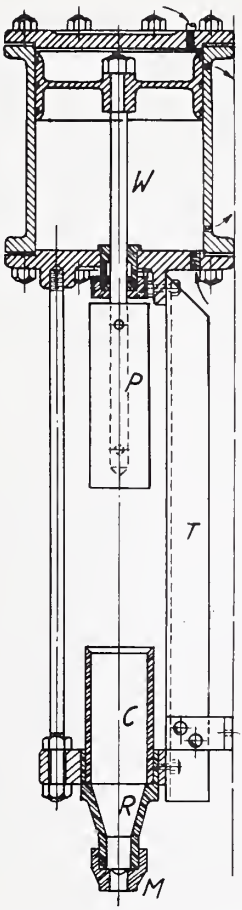
Previous work has shown that the silica content of a porcelain body is an important factor in determining the magnitude of the coefficient of expansion. For the purpose of throwing additional light upon the relation between composition and thermal expansion, a series of bodies was prepared. In addition, other bodies pertaining to a series of other experiments were included in this study.

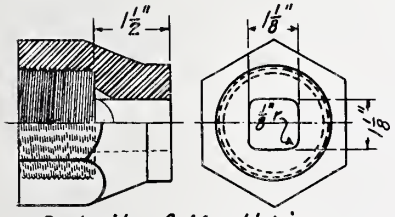
The test specimens employed were rods, 2 cm. in diameter and of such length that when fired to maturity they measured 31 cm. They were pressed in the stiff-plastic state in a small hydraulic plunger press, which was found to be very satisfactory for this purpose, inasmuch as the rods were remarkably free from laminations. The construction of the press is shown in Fig. 1. The fired bars were forwarded to the Expansivity

[1] By permission of the Director, Bureau of Standards.

[2] Watts, *Trans. Am. Ceram. Soc.*, **13**, 406 (1911); Purdy and Potts, *Ibid.*, **13**, 430 (1911); Boeck, *Ibid.*, **14**, 470 (1912); Purdy, *Ibid.*, **15**, 499 (1913); Bleininger and Riddle, *J. Am. Ceram. Soc.*, **2**, 564 (1919).







Detail of Mouthpiece

W - C.I. Cylinder 10" diam.
P - Plunger 3½" × 11⅛"
T - 10" Channel.
C - Clay Cylinder 3½" × 8"
R - Reducer 3½" to 1½" diam.
M - Mouthpiece

FIG. 1.

Section of the Bureau of Standards at Washington, where their coefficients of thermal expansion were determined for the temperature ranges 25–200°, 200–400° and 25–400° C. It would have been desirable to make this determination also at 600° but the use of the apparatus adapted to this temperature involved the expenditure of more time than was available during the period when this work was done.

In a study of the thermal expansion of porcelain it should be understood that a low coefficient, in general, implies a more satisfactory body than a higher value, from the standpoint of mechanical resistance to sudden temperature changes; just as glass of the Pyrex type, with a low thermal expansion, is superior to ordinary glass. In addition, it is desirable that the coefficient should not be subject to decided variations at different temperatures. Thus, a composition having a comparatively small thermal expansion at one temperature, yet a considerably higher one at another temperature but several hundred degrees removed, is not as desirable as one in which the function is a more nearly constant one.

Variations in the value of the coefficient at different temperatures are always suggestive of possible molecular changes or transformations of one or more constituents of the porcelain, whether it be that of alpha to beta quartz, of alpha to beta cristobalite, or inversions as yet undetermined. In case cristobalite has been formed it should manifest itself in a volume change occurring at about 230° C. The effect of the quartz transformation could not be detected in the present work owing to the lower temperatures employed.

The first series of bodies to be considered included compositions which were studied with reference to their possible use as chemical porcelains and whose clay contents varied from 70 to 85 per cent. The content of flint was low and varied inversely with the percentage of feldspar. In no case was more than 50 per cent of raw kaolin used. The balance of the kaolin was introduced in the calcined state (cone 14). The constituents of these bodies together with their coefficients of thermal expansion are given in Table 1. Their chemical compositions are shown in Table 2. The clay content was increased from 70 to 85 per cent

TABLE 1.

Composition of Bodies Used and Their Coefficients of Expansion.

Body No.	51	52	53	54	55	56	57	58	59	60	61	62	63
% Raw kaolin	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0
% Calcined kaolin	20.0	20.0	20.0	20.0	20.0	25.0	25.0	25.0	25.0	30.0	30.0	30.0	35.0
% Total clay	70.0	70.0	70.0	70.0	70.0	75.0	75.0	75.0	75.0	80.0	80.0	80.0	85.0
% Flint	18.5	13.5	9.5	5.0	....	13.5	10.0	5.0	....	8.5	5.0	....	....
% Whiting	10.0	15.0	19.0	23.5	28.5	10.0	13.5	18.5	23.5	10.0	13.5	18.5	13.5
% Feldspar	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Coefficient of Expansion $\times 10^{-6}$.													
25–200° C.	6.1	3.4	3.3	3.7	2.3	4.1	3.7	3.2	3.5	4.7	3.3	2.9	3.2
200–400° C.	5.1	3.6	4.4	5.3	3.0	4.1	4.3	3.7	4.5	4.6	3.2	4.0	3.5
25–400° C.	5.5	3.5	3.7	....	3.1	4.1	4.0	3.4	4.0	4.7	3.3	3.5	3.3

TABLE 2.

Chemical Compositions.

SiO_2	64.59	62.90	61.61	60.10	58.52	62.07	60.90	59.27	57.64	59.56	58.36	56.80	55.93
Al_2O_3	30.86	31.76	32.53	33.36	34.25	33.15	33.80	34.70	35.63	35.43	36.08	37.00	38.40
TiO_2	0.15	0.15	0.15	0.15	0.15	0.17	0.17	0.17	0.17	0.18	0.18	0.18	0.19
Fe_2O_3	0.53	0.55	0.57	0.61	0.63	0.56	0.58	0.60	0.64	0.63	0.65	0.62	0.66
CaO	1.07	1.08	1.09	1.10	1.12	1.09	1.11	1.15	1.14	1.10	1.12	1.13	1.13
MgO	0.22	0.23	0.23	0.24	0.25	0.23	0.24	0.25	0.25	0.26	0.27	0.28	0.28
K_2O	1.58	2.08	2.47	2.92	3.40	1.61	1.97	2.45	2.96	1.67	2.05	2.53	2.07
Na_2O	1.05	1.25	1.35	1.52	1.68	1.12	1.23	1.41	1.57	1.17	1.29	1.46	1.34
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Ratio: $\frac{\%\ \text{Silica}}{\%\ \text{Alumina}}$ =	1:2.09	1:1.97	1:1.9	1:1.8	1:1.71	1:1.87	1:1.8	1:1.71	1:1.61	1:1.68	1:1.61	1:1.53	1:1.45

and the flint content decreased from 18.5 to 0 per cent. The silica content therefore decreases gradually from 64.59 to 55.93 per cent. Owing to this comparatively small variation in silica content, there are no marked variations in the coefficients of expansions.

As a whole, it may be said that there is a tendency for bodies high in clay substance to show lower thermal expansions. But it is apparent from the data that bodies high in clay substance have low thermal expansions, fairly independent of the small variations in quartz content which are possible. It is evident that the case is somewhat different with bodies containing 70 per cent or less of clay substance and therefore a silica content of more than 64 per cent. From this point the thermal expansions increased appreciably. This is illustrated by the following two porcelain bodies of normal clay content:

	Body No. 24.	Body No. 220.
Clay	50.0%	50.0%
Flint	34.0	32.5
Feldspar	16.0	16.0
Whiting	0.0	1.5
Coefficient $\times 10^{-6}$ at		
25–200° C.	4.6	6.2
200–400° C.	4.7	4.6
25–400° C.	4.6	5.3

A second series of porcelain bodies was planned to show the effect of replacing flint by synthetic sillimanite ($Al_2O_3.SiO_2$), a material more constant in volume with reference to the thermal expansion. Some of the same bodies given before were included, *i. e.*, Nos. 51, 56, 60, 63 and 220.

The compositions and ultimate chemical analyses of the bodies are given in Table 3. It will be noted that 30 per cent sillimanite was introduced into bodies Nos. 221, 216, 217, 218 and 219. It was substituted for all of the flint and the balance, if any, took the place of calcined clay. In one case, body 221, there was an excess of flint, 2.5 per cent, still remaining after the sillimanite had been added.

The effect of the substitution is shown in Table 4. For convenient reference the contents of clay substance, flint and sillimanite are given together with the coefficients of expansion.

TABLE 3.

Body No.	51	216	56	217	60	218	63	219	220	221
% Raw clay	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0
% Calcined clay	20.0	8.5	25.0	8.5	30.0	8.5	35.0	5.0	....	....
% Flint	18.5	....	13.5	....	8.5	....	....	....	32.5	2.5
% Sillimanite	....	30.0	....	30.0	....	30.0	....	30.0	....	30.0
% Feldspar	10.0	10.0	10.0	10.0	10.0	10.0	13.5	13.5	16.0	16.0
% Whiting	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
% Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Chemical Compositions.										
SiO_2	64.59	49.81	62.07	49.81	59.56	49.81	55.93	50.38	72.51	52.10
Al_2O_3	30.86	45.05	33.15	45.05	35.43	45.05	38.40	44.10	22.60	42.24
TiO_2	0.15	0.24	0.17	0.24	0.18	0.24	0.19	0.23	0.16	0.22
Fe_2O_3	0.53	0.64	0.56	0.64	0.63	0.64	0.66	0.63	0.44	0.59
CaO	1.07	1.11	1.09	1.11	1.10	1.11	1.13	1.13	1.05	1.10
MgO	0.22	0.28	0.23	0.28	0.26	0.28	0.28	0.27	0.18	0.26
K_2O	1.58	1.67	1.61	1.67	1.67	1.67	2.07	1.98	1.98	2.18
Na_2O	1.05	1.20	1.12	1.20	1.17	1.20	1.34	1.28	1.08	1.31
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Ratio: $\frac{\%\ \text{Silica}}{\%\ \text{Alumina}}$ =	1:2.09	1:1.11	1:1.87	1:1.11	1:1.68	1:1.11	1:1.45	1:1.14	1:3.21	1:1.23

TABLE 4.

Body No	220	221	51	216	56	217	60	218	63	219
% Raw clay	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0
% Calcined clay	....	....	20.0	8.5	25.0	8.5	30.0	8.5	35.0	5.0
% Total clay	50.0	50.0	70.0	58.5	75.0	58.5	80.0	58.5	85.0	55.0
% Flint	32.5	2.5	18.5	....	13.5	....	8.5	....	....	....
% Sillimanite	....	30.0	....	30.0	....	30.0	....	30.0	....	30.0
Coefficients of Expansion $\times 10^{-6}$.										
25–200° C	6.2	2.9	6.1	3.1	4.1	3.1	4.7	3.1	3.2	3.4
200–400° C	4.6	4.0	5.1	3.8	4.1	3.8	4.6	3.8	3.5	3.9
25–400° C	5.3	3.5	5.5	3.5	4.1	3.5	4.7	5.5	3.3	3.7
Ratio: $\frac{\%\ \text{Silica}}{\%\ \text{Alumina}} =$	1:2.09	1:1.11	1:1.87	1:1.11	1:1.88	1:1.11	1:1.45	1:1.14	1:3.21	1:1.23

It will be noted that as sillimanite replaces flint in the bodies lower in clay content the reduction in thermal expansion is much more marked than in those bodies in which the clay content is high. As the content of clay increases the difference becomes smaller. The final body, with 85 per cent of clay, is not improved by the sillimanite substitution. It is obvious, of course, that in such compositions the possible content of flint can only be small. There can be no doubt but that the flint is the greatest factor in causing increased thermal expansion, not only by virtue of the properties of the crystalline quartz, but also through the increase of the silica content of the body, *per se*.

Upon replacing the flint by any substance, more inert from this standpoint, whether it be calcined kaolin, sillimanite or some other material, the general effect is a decrease in the thermal expansion.

Conclusion.

Bodies high in clay substance show low thermal expansions. Variations in flint content within the narrow limits possible in such bodies do not indicate any decided lowering of the expansivity. As the clay content of porcelains is lowered and the amount of quartz becomes larger, its quantitative effect as regards thermal expansion becomes more marked. Any replacement of quartz by substances more inert from this standpoint will lower the expansivity and calcined kaolin may be as effective in this respect as sillimanite or other synthetic silicates. Bars made of the synthetically prepared sillimanite and burned to cone 26 showed lineal thermal coefficients of expansion as follows: 25–200° C = 2.7×10^{-6}, 200–400° C = 3.9×10^{-6} and 25–400° C = 3.3×10^{-6}.

Bureau of Standards,
Pittsburgh, Pa.

FURTHER STUDIES ON PORCELAIN.[1]

By F. H. Riddle.

Previous work in this laboratory, concerned with the study of porcelains,[2] dealt with the consideration of the firing ranges of various compositions. Porcelain bodies of widely varying compositions were examined but the methods of preparation were kept constant throughout the work. The present study includes further variations in composition and also deals with different methods of preparation.

Effect of Small Additions of MgO and CaO.—The effect of introducing small quantities of the alkaline earths, including BaO, has been discussed at length in the article mentioned above. Considerable evidence is also available from the work of others.[3]

The present work was intended to trace the effect of the CaO and MgO upon the microstructures of the porcelains.

The base composition was as follows:

	Per cent.
Flint	37
Potash feldspar	18
Ball clay	6
Kaolin	39
	100

The kaolin consisted of equal parts of Florida, Georgia, N. Carolina and Delaware. The ball clay consisted of equal parts of Tennessee No. 5 and Kentucky No. 4. The fluxes were added for the purpose of lowering the maturing temperatures of those bodies containing a low percentage of feldspar as the only flux.

[1] By permission of the Director, Bureau of Standards.

[2] *J. Am. Ceram. Soc.*, 1, 606 (1918).

[3] A. E. Hottinger, *Trans. Am. Ceram. Soc.*, **5**, 132 (1903). L. E. Barringer, *Ibid.*, **6**, 86 (1904). E. Ogden, *Ibid.*, **7**, 370 (1905). C. E. Jackson, *Ibid.*, **8**, 114 (1906). A. S. Watts, *Ibid.*, **10**, 265, 543; and **11**, 185 (1909). E. Orton, Jr., *Ibid.*, **10**, 543 (1908). H. Hope, *Ibid.*, **11**, 494 (1909). A. V. Bleininger and R. T. Stull, *Ibid.*, **12**, 628 (1910). L. Ogden, *Ibid.*, **13**, 395 (1911).

Although the fluxing value of feldspar cannot be considered equivalent to that of the alkaline earths used, for the sake of simplicity it was replaced by equal amounts of the other fluxes.

The four bodies examined petrographically were:

No. 164, containing 0.2 per cent MgO.
No. 167, containing 1.0 per cent MgO.
No. 168, containing 0.2 per cent CaO.
No. 171, containing 1.0 per cent CaO.

These percentage additions were, of course, not chemically equivalent—since the molecular proportion of MgO was greater than that of CaO.

Fig. 1 shows the temperature-porosity ranges of the extremes

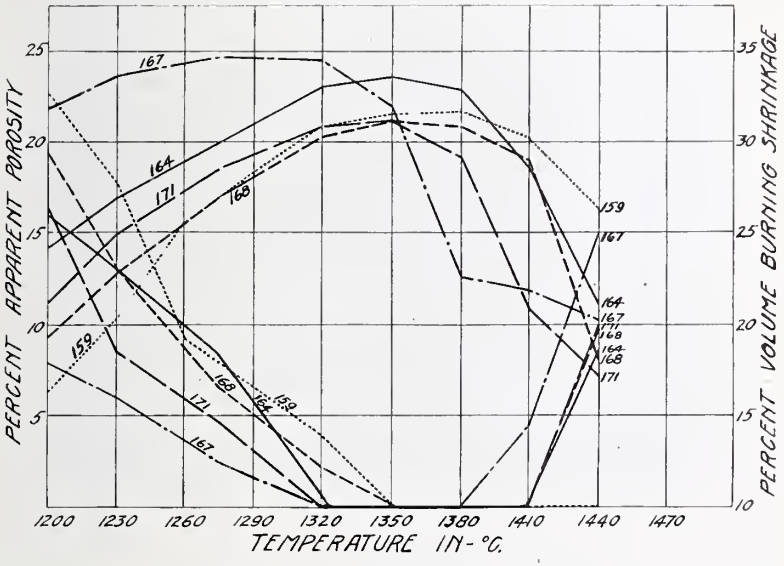


Fig. 1.

of the MgO- and CaO-series together with similar curves for body No. 159, having the same composition as the others but lacking the alkaline-earth fluxes. Without these additional fluxes the body requires a higher temperature to reach maturity. It will also be noted from the diagram that the maturing tempera-

ture of the body containing MgO is lower than that of the body containing CaO.

The five bodies in question show wide differences when examined microscopically in thin sections. The specimens examined under the microscope were fired together to the softening temperature of cone 14. Although this temperature is higher than is required to mature the bodies containing the alkaline earths, the sections show quite markedly the effect of the additions. From the photomicrographs of the sections (Figs. 2, 3, 4, 5 and 6), it appears that solution of the quartz grains has taken place to a varying extent and that the amount, distribution and size of the sillimanite crystals likewise show distinct differences.

The section of body 159, Fig. 2, indicates some solution of the

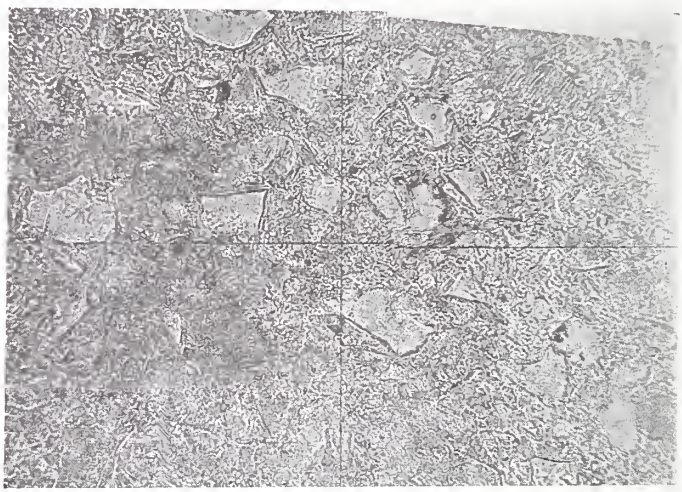


FIG. 2.—Body 159, cone 14 (200 diameters).

quartz grains around the corners and edges but it has not gone very far. The clay is entirely dissociated and the large original grains have become aggregates of numerous, long, slender crystals of sillimanite. The great majority of the crystals are smaller than 9μ (0.009 mm.), scarcely discernible as definite crystals. Some crystals of the size 9μ are to be found and a few between

9μ and 18μ. The glassy ground-mass has a cloudy appearance, presumably due to very minute crystallites of sillimanite derived from clay dissolved in the feldspar. The larger quartz grains show narrow zones of glass surrounding each particle.

From the section of body 164, Fig. 3, it appears that the struc-

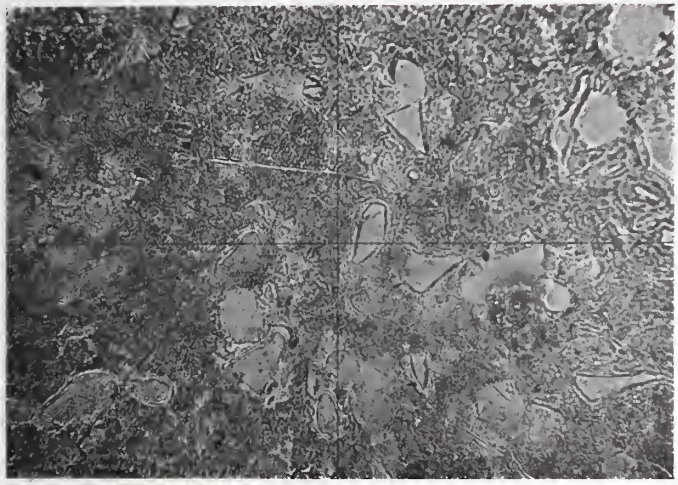


FIG. 3.—Body 164, cone 14 (200 diameters).

ture is decidedly more glassy and the ground-mass apparently more homogeneous. Large aggregates of sillimanite crystals are infrequent. The crystals have become thicker but their lengths remain about the same. No great change is to be expected since the flux in the body probably does not penetrate the larger clay grains but simply dissolves their outer portions. The greater homogeneity of the ground-mass has been brought about by the nearly complete solution of the larger clay grains. Although in body 159 the sillimanite crystals in the glassy ground-mass were too minute for measurement, they have now increased in size until crystals 5μ in size are quite frequent. Many remain which are smaller and hardly measurable. There is a pronounced difference to be noted as regards the quartz grains, which show a general rounding of the corners as compared with the angular

appearance of the quartz in body 159. Minute quartz grains have disappeared.

The photomicrograph of body 167, Fig. 4, shows still more

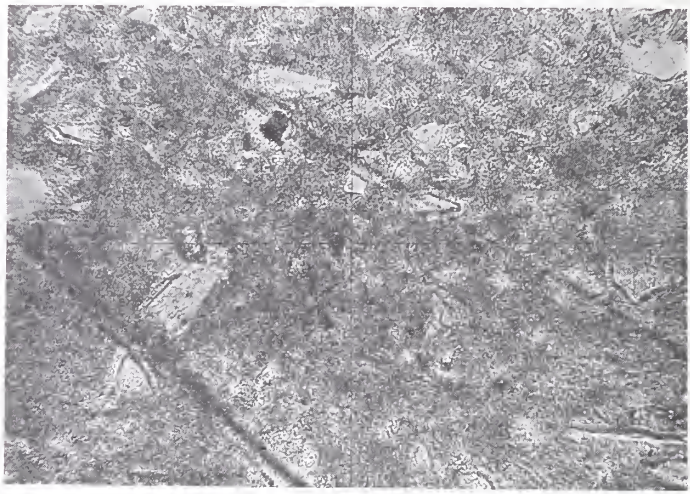


FIG. 4.—Body 167, cone 14 (200 diameters).

extensive solution of the quartz. The same general lack of aggregates of sillimanite crystals is observed as in body 164, the groundmass is more glassy and the crystals imbedded in it are larger. The sillimanite crystals range in size from 21 μ to 27 μ in length down to sizes too small for measurement. The average length is about 11 μ. Some of the larger crystals are sufficiently thick to show interference colors. Frequently the interlocking of the crystals takes on the peculiar lattice structure noted by Klein.[1] While the size of the crystals has increased their number has decreased. To this is due, apparently, the more glassy appearance of the section. The greater translucency of the body is probably due to this and also to the increase in glass, due to quartz-solution.

The microstructure of body 168, Fig. 5, is much the same in

[1] Bur. Standards, *Tech. Paper* 80.

appearance as body 164. Quartz-solution appears to have progressed further since the glassy zone is somewhat wider.

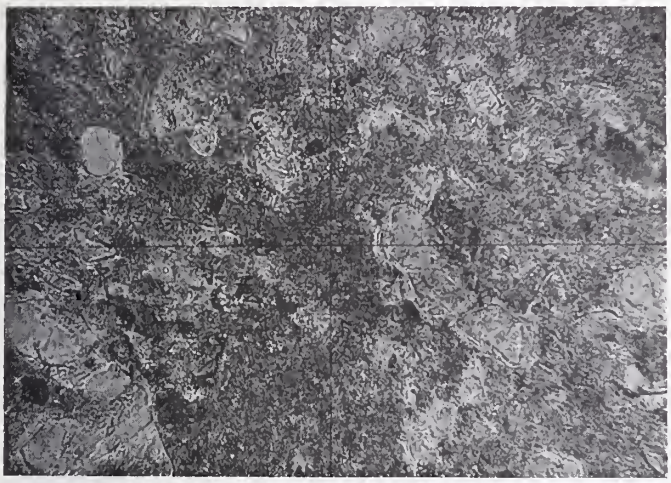


FIG. 5.—Body 168, cone 14 (200 diameters).

The section of body 171, Fig. 6, corresponds closely to that of body 167. Quartz-solution is slightly greater. The sillimanite crystals are a little larger, averaging 14μ.

It should be noted that both bodies, 167 and 171, are overfired. When burned to cone 11 they show considerable sillimanite formation but only a comparatively limited solution of quartz. They did not appear to have as good a structure as body 159 at cone 14, lacking in homogeneity. The bodies containing the alkaline-earth fluxes seem to be made up of an aggregate of quartz grains and decomposed clay substance cemented together by the glassy ground-masses.

The translucency of the bodies containing the MgO and CaO additions is as great at cone 11 as that of the body with only feldspar at cone 14, indicating that the amount of glass formed is increased.

A comparison of bodies 159, 168 and 171 shows that the use of CaO, like MgO, increases the sillimanite formation as well as the solution of the quartz, this being very probably due to the decreased viscosity of the body.

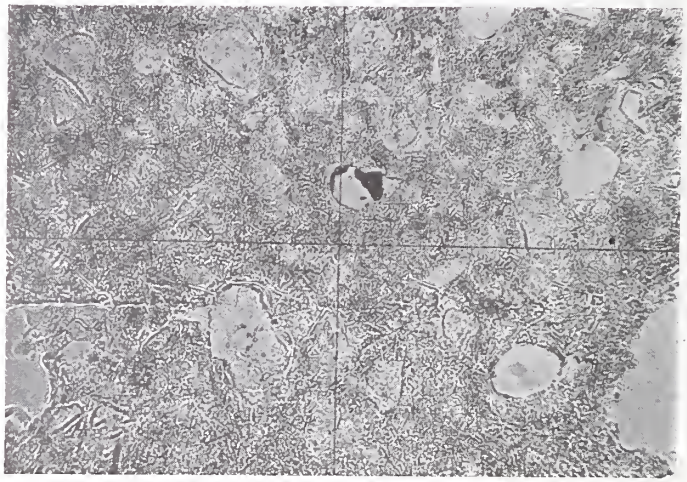


FIG. 6.—Body 171, cone 14 (200 diameters).

This observation differs from that of Watts,[1] although it must be understood that the bodies used were not the same nor, very likely, the conditions of firing.

Comparison between the Effect of Whiting and Dolomite.—It has been found that the addition of small amounts of dolomite to porcelains, maturing between cones 9 and 11, is effective in bringing about earlier vitrification. The action of dolomite is somewhat different from that of whiting and it is of interest to note this difference.

For the purpose of this part of the study a body somewhat similar to the type in use by American manufacturers of vitreous tableware was employed. Its composition was as follows:

[1] *J. Am. Ceram. Soc.*, **2**, 400 (1919).

	Per cent.
Flint	36
Potash feldspar	16
Tennessee ball clay No. 5	6
Florida kaolin	10
Delaware kaolin	9
English china clay	20
Dolomite or whiting, or both	3
	100

The only variation in the body was in the proportion of whiting to dolomite making up the total of 3 per cent. The proportions added were as follows:

TABLE 1.

No. of body.	Whiting. Per cent.	Dolomite. Per cent.
222	3.0	0.0
223	2.5	0.5
224	2.0	1.0
225	1.5	1.5
226	1.0	2.0
227	0.5	2.5
228	0.0	3.0

It is evident, of course, that these proportions are not molecularly equivalent, since the dolomite end of the series carries proportionately more flux than the whiting end.

The test pieces made from these bodies were fired at one time in a large gas fired test kiln, at a rate practically the same as that employed in firing commercial bisque-kilns. The temperature reached was that indicated by cone 11 and care was taken to prevent too rapid cooling. Discs were also made of the bodies, bisqued to cone 1, glazed, and fired to cone 11 down.

The seven fired bodies were subjected to tests for the purpose of determining their color, translucency, toughness, absorption, porosity, microstructure and crushing strength. The size and shape of the specimens needs no description. It might be stated, however, that for the estimation of the toughness of the bodies, pieces measuring 2″ in length and $^7/_8$″ in depth and width, in the fired state, were used.

Color.—All of the bodies were of excellent color and it would

be difficult to state definitely which end of the series was superior in this respect. Perhaps the color of the dolomite end was slightly richer in tone than the whiting end of the series.

Translucency.—All bodies, when made up into saucers of 2 gage thickness and fired (glazed) at cone 11, were very translucent. There was very little difference in this respect, although the dolomite end of the series showed somewhat greater translucency. All of the bodies had a fine appearance.

Toughness.—This quality was determined by means of the rattler test which has been used by others[1] but for which no standard has been adopted. The specimens used by us were made with square edges and corners and were of such size that they could be tested in an ordinary ball mill. The sharp edges were found to be a very desirable feature, inasmuch as they afforded an opportunity for the chipping of brittle bodies during the first half-hour of the run. This loss in weight is very significant and more representative of the actual behavior of the body than the total loss after a run of longer duration, since, after the corners are worn down, the specimens are worn down more by attrition than by impact. For this reason rounded specimens are not desirable. For table ware we are more interested in toughness, as evidenced by resistance to chipping, than in resistance to abrasion.

The rattler tests were made in a ball mill, $9^3/_4''$ in diameter and 13″ long, inside, rotating at a speed of 40 revolutions per minute. The charge was made up of 61 pebbles of nearly uniform size, weighing 22 pounds and 12 ounces.

For each test, 13 porcelain specimens were used. If the tests were to be standardized it would, of course, be advisable to use, say, 10 kg. of pebbles and 12 specimens. The results of the tests made on bodies 222 to 226 are comparable throughout since the same conditions were maintained. The mill was stopped at time intervals of 15, 30, 60, 120 and 180 minutes and the specimens weighed. The losses in weight, expressed in per cent of the original weights of the charges, are given in the following tables:

[1] Watts, *Trans. Am. Ceram. Soc.*, **1**, 86 (1899). E. Ogden, *Ibid.*, **7**, 370 (1905).

TABLE 2.

Percentage Loss.

Body No.	15 Minutes.	30 Minutes.	60 Minutes.	120 Minutes.	180 Minutes.
222	4.67	6.38	7.86	9.86	11.25
223	4.09	5.60	7.72	10.45	11.40
224	4.47	5.98	9.20	11.10	12.80
225	2.95	4.60	7.00	8.80	9.42
226	3.66	5.20	7.00	9.45	10.70
227	3.60	5.15	6.85	8.70	9.80
228	3.05	4.12	5.80	8.40	10.30

The losses in weight in rattling are also shown in the curve of Fig. 7. It will be noted that as a whole the relations are fairly

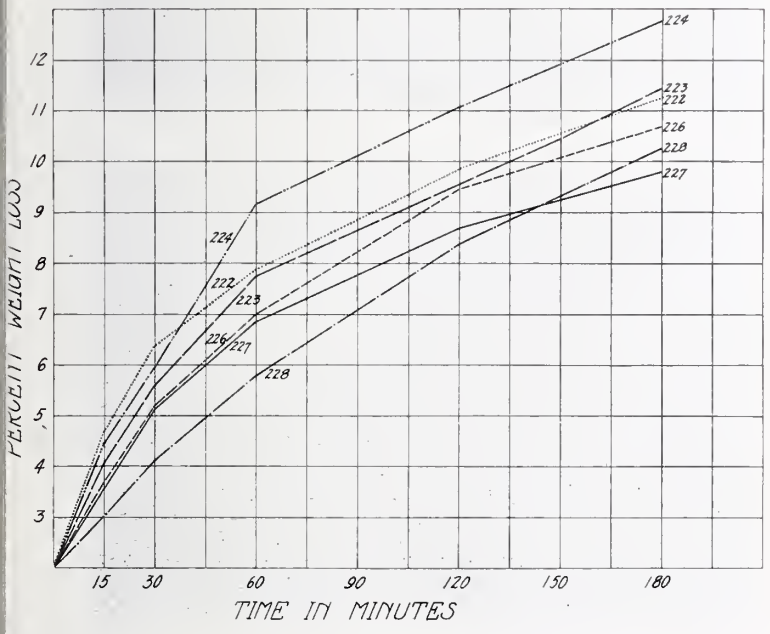
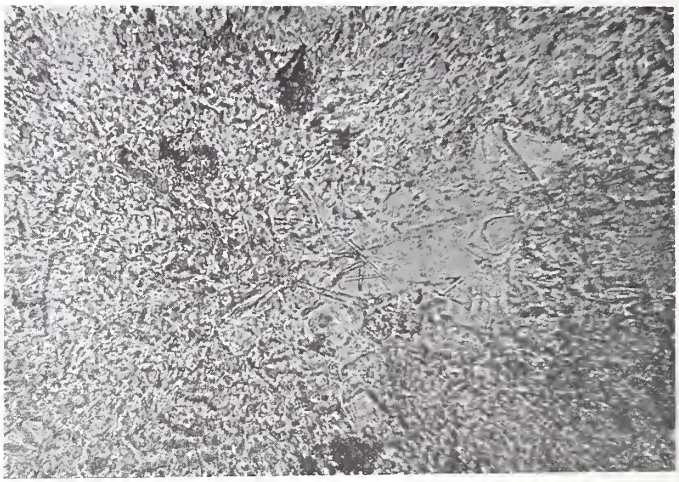


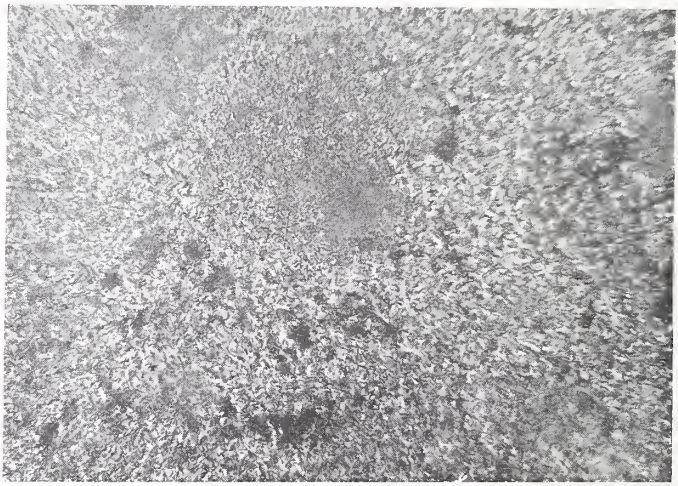
FIG. 7.

consistent. No great differences are to be found after the first part of the run nor at the end of the test. It is evident, therefore, that the toughness is nearly of the same magnitude for all

of the bodies. Still, the high-whiting end of the series shows more loss due to chipping than the dolomite end and, with the



Body 228.



Body 222.

FIG. 8.—Cone 11 (300 diameters).

exception of body 225, the loss decreases almost directly with the decrease in whiting. While this evidence tends to show that dolomite is productive of greater toughness it also seems

apparent that the lime bodies are somewhat harder, since they were worn less after the chipping stage during the first part of the run had been passed. This is noticeable from the gradual dropping off of the loss curve, particularly of the curves for bodies 222 and 228. The variation is not sufficiently striking to warrant making a positive generalization.

Microstructure.—Inspection of the bodies showed that the higher the content of whiting the more dense and vitreous the structure appeared to be. The high-dolomite end appears to be much more crystalline and breaks with a rough fracture, like sugar.

The characteristic structure may be compared by examining the microsections of bodies 222 and 228, representing the whiting and dolomite ends of the series, respectively (see Fig. 8).

Considering the fact that these bodies were fired to cone 11, it is noted that the solution of the quartz and the formation of sillimanite are well advanced. Body 222 shows a more nearly homogeneous structure and comparatively little well-defined crystalline sillimanite, though there undoubtedly is an appreciable amount of the latter present. On the other hand, body 228 shows a considerable amount of well-crystallized sillimanite, evident from the appearance of the coarser fractures and its lower loss in the rattler test. The solution of the quartz is similar in both cases.

Compressive Strength.—Cylindrical specimens of the members of this series were subjected to crushing tests with the following results:

Body No.	Lbs. per sq. inch.
222	57,500
223	38,700
224	49,100
225	55,800
226	46,200
227	55,700
228	55,600
Average,	51,228

It will be noted that the highest strength is obtained with the use of whiting alone, but that the variations in values of this

kind are so great that we can only say that there is no marked differences between the extremes. The crushing values obtained were quite high in all cases and fully equal to strengths of commercial porcelains.

It would seem, therefore, that the use of dolomite in replacing whiting, wholly or partially, is justified, especially in producing bodies of superior toughness or resistance to chipping, such as table ware. Its low cost is an additional point in its favor.

Effect of Fine Grinding.—The question as to the effect of longer or shorter wet grinding is still a more or less mooted one. While some advocate long grinding, others are of the opinion that the best results are obtained with shorter grinding.

The bodies discussed in previous paragraphs were ground for three hours in ball mills $9^3/_4''$ in diameter and $13''$ long, inside, rotating at a speed of 40 revolutions per minute. Five kilograms of charge were ground in each mill. It was thought desirable to study the effect of longer grinding with reference to the effect upon the content of plasticity water, drying and burning shrinkage and porosity changes.

The body selected for this work had the composition:

	Per cent.
Kentucky ball clay No. 4	4.00
Tennessee ball clay No. 5	4.00
Florida kaolin	9.25
Delaware kaolin	9.25
Georgia kaolin	9.25
N. Carolina kaolin	9.25
Feldspar	18.00
Flint	37.00
	100.00

This body when ground for three hours was numbered 159; 6 hours grinding, 160; 12 hours, 161; 24 hours, 162; and 48 hours, 163.

Test specimens were prepared in the usual way and fired to maturity. The water of plasticity and the drying shrinkages, expressed in terms of the dry volumes, were found to be as follows:

TABLE 3.

No. of body.	Per cent water.	Per cent volume drying shrinkage.
159	28.50	15.77
160	28.20	14.20
161	29.63	14.75
162	31.85	15.90
163	34.40	16.73

It is evident that the water of plasticity and with it the drying shrinkage are increased by longer grinding. The difference is not marked up to 24 hours, but after that it becomes quite apparent. It is quite probable that grinding for too long a time is very apt to give a body of unsatisfactory working behavior, especially in large cylinders where the grinding effect is very much more intense and where even one additional hour may bring about a considerable change.

The porosity and volume changes undergone by the body ground for different periods of time are given in Table 4 and shown graphically in Fig. 9.

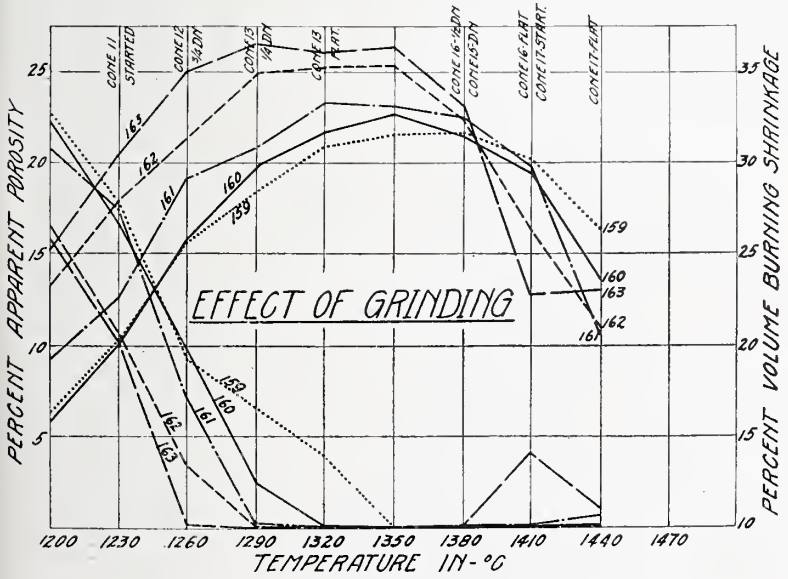


FIG. 9.

TABLE 4.

°C.	Body 159. 3 hrs. grinding.		Body 160. 6 hrs. grinding.		Body 161. 12 hrs. grinding.		Body 162. 24 hrs. grinding.		Body 163. 48 hrs. grinding.	
	Volume shrinkage. %.	Porosity. %.	Volume shrinkage. %.	Porosity. %.	Volume shrinkage. %.	Porosity. %.	Volume shrinkage. %.	Porosity. %.	Volume shrinkage. %.	Porosity. %.
1200	16.48	22.70	15.92	22.20	19.48	20.85	23.30	16.55	25.20	15.82
1230	20.30	17.75	20.15	16.75	22.70	17.45	27.95	10.88	30.50	10.40
1260	25.50	9.36	25.65	9.87	29.10	7.21	28.50	3.50	35.00	0.05
1290	28.40	9.24	29.85	2.39	31.90	0.08	34.95	0.0	36.55	0.0
1310	30.82	4.02	31.60	0.08	33.30	0.0	35.05	0.0	36.00	0.0
1340	31.63	0.0	32.55	0.0	33.18	0.0	35.15	0.0	36.30	0.0
1370	31.65	0.0	31.55	0.0	32.60	0.0	32.35	0.0	33.12	0.0
1410	30.10	0.0	29.30	0.0	29.80	0.0	26.42	0.0	22.87	4.15
1440	26.42	0.0	23.32	0.0	20.60	0.0	20.90	0.0	23.05	1.10

The fact that finer grinding causes earlier maturity and earlier overfiring is brought out very strikingly. The body when ground three hours matured at 1340° C, and the same body, ground 48 hours, reached zero absorption at 1290°, a difference of about 50°. The intermediate grinds mature quite regularly in proportion to the time of grinding. It would seem, then, that the effect of longer grinding is equivalent to an increase in feldspar or other fluxes or in ball clay. It appears also that longer grinding may be practiced with bodies low in ball clay in order to reach the desired degree of maturity which otherwise would require a higher content of this constituent. The petrographic inspection of the bodies ground for different periods of time revealed no essential differences in the microstructures.

The writer is indebted to Mr. A. B. Peck for valuable assistance in the preparation and examination of the thin sections, also to Mr. A. V. Bleininger and members of the Clay Products Section of the Bureau of Standards.

Bureau of Standards,
Pittsburgh, Pa.

THE REHYDRATION OF CALCINED CLAYS.

By J. S. Laird and R. F. Geller.

Introduction.

It has been shown by Mellor,[1] Sokolov,[2] Bleininger,[3] Brown and Montgomery,[4] and others that while clays and kaolins do not appear to have a definite decomposition temperature, there is little loss of water on heating them to temperatures below 500–600° C, but that heating them to higher temperatures for a sufficient time causes complete dehydration. In the case of plastic clays the plasticity diminishes as a rule as they are dehydrated, and disappears completely on heating to a red heat, while a much coarser, granular texture is developed in the clays.

That there is little tendency towards the recombination of water with calcined clay is shown by the prolonged resistance to weathering of even very soft-burned clay products, when not subject to frost action or containing soluble salts. Reported analyses of pieces of very old soft-burned pottery show sometimes as high as 3 per cent water. Calcined clay absorbs from moist air considerable hygroscopic water; according to Stull[5] upwards of 5 per cent in the case of graphite-clay crucibles. This hygroscopic water is driven off by drying at 110–120° C. Hornung[6] has shown that a surface clay, calcined at any temperature below 900° C, when kept in water for two weeks took up small amounts of water not capable of being driven off at 110° C, and, therefore, apparently chemically combined. The amount taken up reached a maximum of 1 per cent in the case of a sample partially dehydrated by being heated to only 350° C. Mellor and Holdcroft,[7] in an investiga-

[1] Mellor and Holdcroft, *Trans. Eng. Ceram. Soc.*, **9**, 94 (1911).

[2] Sokolov, *Z. Kryst. Mineralog.*, **55**, 195–6 (1917).

[3] A. V. Bleininger, Bur. of Standards, *Tech. Paper* **1**.

[4] G. H. Brown and E. T. Montgomery, *Trans. Am. Ceram. Soc.*, **14**, 709 (1912).

[5] R. T. Stull, *J. Am. Ceram. Soc.*, **2**, 209 (1919).

[6] M. R. Hornung, Thesis, University of Illinois.

[7] Mellor and Holdcroft, *Trans. Eng. Ceram. Soc.*, **10**, 169 (1912).

tion of the chemical constitution of the kaolinite molecule, attempted to rehydrate kaolin which had been calcined at 600–640° C, by heating it in water under pressure to 300° C. Under this treatment the kaolin took up only 2.5 per cent water and did not become plastic. They ascribed the water taken into combination to hydration of free silica, and concluded that on dehydration the

$$O = Al - O - Si = O$$

kaolinite did not give the anhydride $\qquad > O$

$$O = Al - O - Si = O$$

but decomposed into free silica and alumina.

In the hope of devising a method of increasing the plasticity of certain non-plastic or slightly plastic clays, we have studied the action of very hot water, necessarily under very high pressures, upon a number of raw and calcined clays and kaolins. We have found that such treatment has little or no effect upon raw clays, but that clays calcined at moderate temperatures can be very largely rehydrated—the rehydrated clay in general showing considerable plasticity.

Experimental.

The treatment of the clays was carried on in high-pressure autoclaves or "vulcanizers," of which the largest had a capacity of about one gallon, and was built to withstand steam pressures up to 1000 pounds per square inch, corresponding to a temperature of about 290° C. The samples treated were in amounts up to several pounds in weight, so that their plasticities, or at least their bonding powers, could be determined by making ordinary tension briquettes and noting their air-dried strengths. The steam pressures were measured by an ordinary pressure gauge, and the temperatures by a mercury thermometer inserted in a well in the cover of the autoclave. The water content of the sample was determined as loss on ignition at 900° C in an electric muffle-furnace, after drying to constant weight in an oven at 110–120° C.

Considerable difficulty was encountered in carrying out the experiments at high pressures, as the autoclaves were not satisfactorily designed for such service. In particular there was difficulty in making a tight gasket-joint between the body and

cover of the autoclave. Soft lead, melted and poured into the gasket groove, formed the most satisfactory packing.

As the action seemed to be a surface one, wet-grinding of samples for periods of 24–48 hours was resorted to in order to decrease the size of the particles and increase the surfaces exposed. It seems probable that the action would be much more rapid if the material could be simultaneously subjected to grinding and treatment in a suitably designed rotary autoclave.

Results.

Pennsylvania Flint Clay:

(A) Coarse lumps of the raw clay were not affected by being heated in water to 200–250° C for a very long time, no slaking being observed after 200 hours. Water-ground samples seemed somewhat more plastic after treatment at 90 pounds for 48 hours.

(B) Calcined at 800° C; loss on ignition 15.05 per cent, residual 0.4 per cent. Coarsely ground samples were apparently not affected by the treatment. After wet-grinding 24 hours and treating in water at 255° C for 30 hours, the clay took up 10.9 per cent water, which was increased by repeating the process to 11.7 per cent. Air-dried strength 52 pounds. When only a moderate amount of water was used with this clay it appeared to set on cooling after removal from the autoclave. This peculiar behavior was not shown by any other clay tested. In order to verify it briquettes of the clay mixed with an equal amount of Ottawa sand were hydrated in the steam space above the water to avoid mechanical disintegration. A tensile strength of 360 pounds per sq. in. was developed on heating to 230° C for 24 hours.

Kittanning Stoneware Clay.—Original water content 8.4 per cent.

(A) Calcined at 600° C one hour; residual water 4.9 per cent; loss 3.5 per cent. Heated in water to 150° C 36 hours; water content 7.5 per cent.

(B) Calcined at 700° C; residual water 1.48 per cent. Heated in water to 230° C 17 hours; water content 7.8 per cent. Heated in water to 230° C 16 hours; water content 6.5 per cent.

(C) Calcined at 800° C; residual water 0.27 per cent. Heated in water to 250° C 48 hours; water content 6.04 per cent. The clay was fairly plastic after treatment.

Florida Kaolin.—Original water content, 13.78 per cent.

Calcined at 600° C; residual water 1.96 per cent. Heated in water to 270° C for 8 hours; water content 11.0 per cent. The clay was fairly plastic after treatment, but rather short and sticky like the raw clay.

Georgia Kaolin.—Original water content 12.06 per cent. Calcined at 700° C; residual water 0.8 per cent.

(A) Heated in water to 270° C for 9 hours; water content 2.34 per cent.

(B) Wet ground and heated as in (A); water content 3.29 per cent.

(C) Heated as in (B) for 9 hours more to 270° C; water content 4.00 per cent.

White-Ware Bisque.—Original water content 0.13 per cent.

Water ground and heated in water to 270° C for 8 hours; water content 0.6 per cent. The material did not become plastic and the coarse-ground material was not slaked by the treatment.

Conclusions.

As a result of the experimental work which has been done we may conclude that:

1. Flint clay cannot be slaked by the action of very hot water in any reasonable length of time, and the plasticities of raw clays are little affected by such action.

2. Clays which have been almost completely dehydrated by calcination at a moderate temperature, 600–700° C, can be rehydrated by being heated in water to temperatures of 200–270° C for a sufficient length of time (8–48 hours). The rehydrated material is in general plastic and appears colloidal. After being worked up and dried two or three times it resembles a raw clay.

3. Clays which have been calcined at higher temperatures can be rehydrated only much more slowly and incompletely.

4. From the ease with which clays dehydrated at a moderate temperature can be rehydrated and brought back to practically their original condition, it seems probable that on dehydration kaolinite forms an anhydride which is broken down by other reactions upon heating to higher temperatures, such as the formation of sillimanite.

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APPARENT VS. TRUE SPECIFIC GRAVITY OF SILICA BRICKS.

By Leon R. Office.

The author, in collaboration with Mr. R. J. Montgomery, submitted a paper at the 1918 meeting of the American Ceramic Society on "Laboratory Testing of Silica Brick."[1] In that paper the importance of the specific-gravity determination was brought out, particularly in control work for the detection of under-burned brick.

Owing to the large amount of silica refractories in a battery of coke ovens, it is at once apparent that it is of the utmost importance to have the bricks and shapes well burned, with as much permanent expansion removed as possible. When the bricks are shipped in large quantities it is necessary to have a method that is both simple and quick to detect under-burning, and at the same time one which can be relied upon. The method employed by us was that of taking a 10-gram chip from the brick and boiling in water for one hour, using a Westphal balance for the weighings. The test required that chips from the same sample check within 0.02.

In a discussion of the author's paper, Mr. D. W. Ross[2] was of the opinion that better results could be obtained by subjecting the specimens to a vacuum while immersed in hot water. Mr. R. M. Howe[3] also presented some interesting data showing determinations under vacuum.

To test out the accuracy of the Westphal-balance method with small chips, several brands of silica bricks were obtained from various parts of the country. These brands covered a range from extremely soft-burned to hard-burned bricks. Two chips were taken from each brick and the apparent specific gravity obtained by careful weighing to the third decimal place. The samples

[1] *J. Am. Ceram. Soc.*, 1, 338 (1918).

[2] *Loc. cit.*, p. 349.

[3] *Ibid.*, p. 346.

were boiled in distilled water for one hour. After its apparent specific gravity had been recorded, each chip was ground in an agate mortar to pass a 100-mesh sieve. All samples, both before and after grinding, were dried at 110° C for several hours and cooled before the tests were made.

The true specific gravity of each powdered sample was obtained

TABLE 1.

Sample No.	Burn.	State.	Apparent sp. gr.	True sp. gr.	Difference.
5984-A	Hard	Penna.	2.265	2.331	+0.066
B	Hard	Penna.	2.285	2.317	+0.032
6094-A	Medium	Penna.	2.461	2.466	+0.005
B	Medium	Penna.	2.461	2.469	+0.008
1997-A	Hard	Penna.	2.320	2.346	+0.026
B[1]	.....	.....	...	...	...
1996-A	Medium	Penna.	2.464	2.467	+0.003
B	Medium	Penna.	2.467	2.471	+0.004
1995-A	Soft	Penna.	2.487	2.482	—0.005
B	Soft	Penna.	2.515	2.511	—0.004
6360-A	Hard	Penna.	2.296	2.329	+0.033
B	Hard	Penna.	2.300	2.321	+0.021
WH-A	Soft	Penna.	2.555	2.557	+0.002
B	Soft	Penna.	2.551	2.557	+0.006
2616-A	Hard	Ill.	2.316	2.316	+0.000
B	Hard	Ill.	2.319	2.321	+0.002
6537-A	Hard	Ill.	2.395	2.417	+0.022
B	Hard	Ill.	2.386	2.411	+0.025
6368-A	Medium	Ill.	2.432	2.437	+0.005
B	Medium	Ill.	2.431	2.436	+0.005
S2-A	Hard	Colo.	2.384	2.384	+0.000
B	Hard	Colo.	2.389	2.392	+0.003
D2-A	Soft	Colo.	2.524	2.528	+0.004
B	Soft	Colo.	2.530	2.529	—0.001
6535-A	Medium	Canada	2.478	2.491	+0.013
B	Medium	Canada	2.492	2.493	+0.001
6536-A	Medium	Canada	2.491	2.494	+0.003
B	Medium	Canada	2.496	2.497	+0.001
					Av., +0.011

[1] Sample destroyed.

by the refined method of Day and Allen.[1] A pycnometer with thermometer and capillary side-arm attached was used. The pycnometer with its charge was placed in water at 80° to 90° C and attached to a suction line for one-half hour to remove entrapped air. Table No. 1 gives the results of the tests with two chips from each brick. The brands are not given but the state in which they are manufactured is indicated. Eight distinct brands and seven manufacturers are represented.

Summary.

Of 27 samples tested 22 showed a slightly higher, two the same, and three a lower true specific gravity.

By taking an average of the differences, the true specific gravity is found to be 0.011 higher than the apparent specific gravity.

If care is taken to obtain two 10-gram chips as representative as possible of the material in a silica brick, and if their respective specific gravities check within 0.02, the result should be very close to the true specific gravity.

Laboratories of The Koppers Company and
Pittsburgh By-Product Coke Company,
Mellon Institute,
Pittsburgh, Pa.

[1] "The Analysis of Silicate and Carbonate Rocks," U. S. Geol. Surv., *Bull.* **422**.

CERAMIC ABSTRACTS.

General.

On the employment of the Brinell ball for testing construction materials. M. M. H. Le Chatelier and B. Bogitch. *La Ceramique*, 371, 17–18 (1919).—The use of the Brinell hardness test for construction materials has certain advantages over the compression test, namely, it is rapid, accurate and more economical than the compression test. For this test a steel ball, 17.5 mm. in diameter, is used with a pressure of 500 kilograms for 1 minute. The diameter of the depression is then measured under a microscope and expressed in mm. The diameter in copper is 4.5 and in lead 10.1. Testing various construction materials by this method gave the following results which check fairly closely:

	Per cent variation.
Fire clay brick—6.0, 6.1, 6.0, 6.1, 6.2.....	3.3
Hard silica brick—5.1, 5.0, 5.1, 5.0, 5.0...	2.0
Tender silica brick—10.1, 10.7, 10.6, 10.6.	3.0
Plaster—10.3, 10.3.....	0.0
Limestone—5.8, 5.8,.....	0.0
Cement mortar—7.0, 7.1.....	1.5

Compression tests made of the same silica brick cut into cubes, 2 cm. on each side, showed a variation which was much greater as follows:

Hard brick—188, 240, 200.
Soft brick—108, 132, 182.

The Brinell test permits a study of the structures of bricks which often varies much on the same brick as follows:

Hard face brick, 4.5, 5.2, 8.3, 6.3, 8.8.
Soft face brick, 8.4, 6.8, 10.4, 8.3, 12.0.

Tests made on different bricks varied as follows:

	Piston side.	Bottom side.
Brick No. 1.....	5.4	5.6
Brick No. 2.....	5.6	5.6
Brick No. 3.....	5.9	6.3

The Brinell testing machine has an advantage in that it is portable and permits of direct control of products in their manufacture.

H. G. Schurecht.

The casting of heavy clay pottery. B. J. Allen. The Ceramic Society. *Pottery Gazette*, 489–496 (1919).—Casting by means of cored molds produces

hollow castings and poor structure. By resorting to easting under pressure, by means of molds with air-tight easings and connected to an air pump, capable of removing the air from the space between the plaster and the casing, the clay could be kept in eontaet with the plaster and the water extraetion from the slip be made to continue until the desired result was obtained. Methods were described for applying pressure to the slip-supply in produeing pieces of unusual thiekness and pieees with tapering sides and it was shown how, by application of pressure to the slip, the slip could be applied to the thick parts before the thinner parts were hard. A mold of this type with a core may be used for lining the insides of pots with speeial materials, *e. g.*, zirconium, by first dipping the core in a slip of zireonium. For casting eovered pots the vacuum method is indispensable to perfeet ware.

H. G. SCHURECHT.

Tunnel oven development in the potteries. By a MEMBER OF THE STAFF. *The Pottery Gazette* 714–717 (1919).—English potters are coming to the eonclusion that tunnel-oven firing is likely to offer a solution of some of their difficulties, espeeially in saving fuel. A tunnel kiln at La Louiviére, Belgium, which is 210 feet long, is described. Trucks of glost ware pass two at a time at the rate of five feet every 35 minutes. This kiln has been in operation continuously for $2^1/_2$ years without once stopping for repairs. A manufacturer at Stoke-on-Trent who is using a Dressler kiln gives the following data: 30 tons of fuel are used per week to effect an output equivalent to 6 ordinary round periodic kilns which utilize 81 tons. Economy in labor amounts to 23 pounds per week. Truck repairs, etc., cost 15 pounds as compared with 49 pounds necessary to keep up sagger repairs, etc., in periodic kilns. The Marlow tunnel kiln of the muffle type is deseribed. A perforated tunnel, at a certain distance from the firing zone, is covered with a second arch, a cavity being left between the two. The hot air rises after passing the hottest zone and is returned into a heat conduit or combustion ehamber, thereby reducing the fuel consumption $33^1/_3\%$. H. G. SCHURECHT.

The English Ceramic Society. *The Pottery Gazette*, No. 506, 836–840 (1919).—At a meeting of the Refractory Section of the Ceramic Society held at Middlesbroug the following papers were read:

"The Geology of the Refractory Materials of the North of England," Atkinson and Stobbs. 22% of the fire clay used in England comes from the north of England.

"Mica Schists for Lining Cupolas and Steel Converters," P. Boswell. Certain roeks of the mica schist group from near Philadelphia are employed as a furnace lining in a large number of American foundries. In the discussion which followed it was stated that in using natural products for furnace linings minimum spalling and chipping were obtained by placing the foliated planes in one particular direction.

"The Corrosion of Coke Oven Walls," W. J. Rees. Sodium sulphate and sodium chloride in coal may cause corrosion of oven walls. These and other salts may be removed by washing the coal.

"Some Criticisms by a Fire Brick Manufacturer," G. Chance. Fire brick users are unreasonable in the following ways: (1) Low prices are demanded rather than quality. (2) Users do not give the manufacturer sufficient time to make brick properly. (3) Users will not try a new brand of brick. (4) Some users like to see iron specks on brick while others do not.

"Factors Influencing the Properties of Silica Brick," Dr. Scott. Effect of magnesia, alumina, iron oxide, iron oxide plus carbon, and titanic oxide were studied. Smallest after-expansions were obtained by using a bond of lime and iron oxide, while the lowest expansions were obtained by using these oxides separately.

"Note on a Silica Brick from a Furnace Row," J. E. Stead. A brick 3" wide before it was put in an open hearth furnace measured $2^1/_2$" at one end and $2^3/_8$" at the other end after it had been in use.

"A New Type of Recuperative Furnace," Walter Rosenhain and E. Coad-Pryor. An experimental furnace for melting glass and for firing refractories at high temperatures was described. The only form of recuperator furnace giving promise was one consisting of a nest or battery of tubes made of a silicon carbide and china clay mixture, through which the incoming air could be drawn while the products of combustion circulated about the exterior tubes. Fire-clay tubes would not stand the test.

"Further Notes on Zinc-Furnace Refractories," J. A Audley. Mülhaeuser was extensively quoted and according to this writer it was shown that the gradual vitrification of parts of muffle walls (especially bottoms) was accompanied by a decrease in refractoriness and resistance to sudden temperature change, but increased the strength, elasticity and extensibility, so that the average life of the muffles was sixty-three days. The use of zirconia as a coating was referred to, but the small contraction of this material would make it hard to fit a body. Applying the zirconia by putting on a number of coatings, each containing an increased amount of zirconia, is suggested. H. G. Schurecht.

Zirconia, its utilization as a refractory, an opacifier and an abrasive. M. A. Granger. *Mineral Foote Notes, 3*, Nos. 7 and 8 (1919).—Brazilian zirconia deposits at Pocas de Caldas are very extensive and contain 73–74% zirconium. Softening point of pure zirconia is 3000° C. Even with 1.25% silica and iron oxide it does not fuse below 2500° C. The thermal conductivity of the fused pure material is very low. Its coefficient of distillation is low (8.4×10^{-7}), which compares favorably with that of carborundum (7.58×10^{-6}) or alundum (7.10×10^{-6}). It is very resistant to abrupt changes of temperature and is chemically inert. Zirconium carbide forms easily when zirconia is heated to a high temp. in contact with carbon the crucibles often becoming destroyed thereby. Specific conductivity of zirconia is 0.0008 at 1200° C and 0.0034 at 1400° C. Dr. Rieke (*Sprechsaal*, 1908, 214), published the first researches on the use of zirconia as a refractory. The following methods for purifying zirconia are described: (1) Treatment with hydrofluoric

acid; (2) Transformation into carbide; (3) Treatment with sulphuric acid or bisulphates; and (4) Alkaline fusion. For practical purposes treatment with hydrochloric acid is sufficient. In the manufacture of zirconia crucibles zirconium hydrate has been tried as a binder. The vessels, however, were too fragile and another binder was sought. Starch intimately mixed with zirconium hydrate and allowed to dry in air until it contained 9–10% zirconia was especially desirable. Moistened plaster-molds, having a wooden core covered with foil, could be used to mold crucibles. Two hours after molding crucibles are removed from mold, dried, and finally burned to 2000–3000° C in an electric furnace. Experiments in Germany showed that a lining of zirconia lasts a long time, and that in spite of the higher initial cost there was a saving of 50%. In metallurgy zirconia could also be employed in the preparation of copper and its alloys. Silicate of soda and tar are used with zirconia in forming the linings in furnaces. Lime may also be used in small quantities with zirconia, giving good results. Zirconia for laboratory use may be purified as follows: After a preliminary treatment with HCl the mineral is heated on a sand bath with H_2SO_4. The solution is evaporated to dryness and taken up with water. Zirconia can then be removed as follows: The addition of potassium sulphate precipitates a double sulphate; the addition of ammonia precipitates hydrated zirconium; the calcination of hydrate or sulphate of zirconium gives zirconia. Zirconia can be used as an opacifier in glazes. Although it increases the cost of a glaze it produces one which is very acid resisting. Because of its hardness zircon has been used as an abrasive for grinding mother-of-pearl. Grindstones for this purpose are not fired but cemented together. H. G. SCHURECHT.

A visit to a Lancashire pottery. By a MEMBER OF THE STAFF. *Pottery Gazette*, No. 506, 828–830 (1919).—The Heaton Mersey Pottery near Manchester is described. A 12-chamber Hoffman kiln with a very tall chimney is used. The clay is blunged and screened and then one portion is dried in a series of drying kilns heated by exhaust gases from the kilns. Another portion is dried in open-air driers. In summer the clay is allowed to settle to a depth of 5"–6" and taken off once a month. In winter the clay is allowed to settle 18 inches and is left for the whole winter. Clay dried in open-air driers is more plastic than that dried in slip kilns. The ware (flower pots, milk bowls, etc.) is thrown on a steam-driven throwing wheel. When "hard-green" a white engobe is applied. The pieces are then dipped in a blue, galena glaze. H. G. SCHURECHT.

Brick for roads. A. B. SEARLE. *The British Clayworker*, **27**, 102–106 (1919).—The structure of bricks should consist of a porous skeleton filled with a slag-like mass which can be observed under a microscope if thin sections one-thousandth of an inch are made. A section of some of the best and toughest road bricks, after immersion in hydrofluoric acid, yield a felted crystalline mass of impure sillimanite, which possesses great strength and

toughness. To reduce laminations the mouthpiece of the auger machine may be provided with two exits—one to either side of the auger shaft—thus avoiding the lamination due to the central shaft of the pug mill. Recent tests show that the strongest brick after drying were found to be those which had been heated to 240° F in an atmosphere saturated with moisture, and then allowed to dry slowly while the temperature was maintained constant. Brick set damp in the kiln are invariably softer than those which are properly dried. H. G. SCHURECHT.

Clay men make new building material. *Brick and Clay Record,* **55,** 484–487 (1919).—A new company, The American Aggregate Co., Kansas City, has been organized to make a light-weight aggregate from shale for cement. The aggregate is made in a continuous kiln by overburning shale. The temperature of burning is approximately 2000° F and the time required is about 2 hours. The aggregate is cooled slowly after burning in order to retain strength. Concrete with crushed stone aggregate weighs 140–155 lbs. per cubic foot. Where the bloated shale (Haydite) is used it weighs 90–100 lbs. per cubic foot. Tests made show that concrete made of this material is 30–40% stronger than when other aggregates are used. Its use for building dwellings, railroad cars and boats has been tried and found satisfactory—having the advantage of being cheap and resistant to weather. H. G. SCHURECHT.

Geological.

Magnesite, its occurrence and uses. By T. CROOK. *The Mining Magazine,* **20,** 115–120, 175–179, 246–249, 308–311 (1919).—The paper constitutes a reprint of an article entitled "Magnesite as a Raw Material," by T. Crook, of the Imperial Institute. Among the many interesting points considered may be mentioned the following: the mineralogy of magnesite and a classification of magnesites on the basis of texture and composition as follows: (1) Spathic breunnerite, example, Styrian, (2) Spathic magnesite, example, Quebec, Washington and Norwegian; (3) Compact magnesite, example, Grecian, Californian, Italian, Indian (Salem) and Australian; (4) Hydromagnesite, example, Atlin in British Columbia. The term "spathic" refers to the comparatively coarsely crystalline magnesites and breunnerites, the constituent grains of which show cleavage. Analyses which show the range in composition of the various types are given. In this connection it is interesting to note that the material from Washington contains not more than 2% of lime while Quebec magnesite contains a considerable amount of dolomite and ranges up to 10% or more of lime. The various types are considered in detail and with respect to their occurrence both geographically and geologically. Comparisons of types are made and throughout the article are analyses of materials from various localities. In connection with the different types the uses of the raw material are mentioned. E. D. ELSTON.

Biennial Report of the State Geologist (1919). *Missouri Bureau of Geology and Mines.*—The report contains brief notes on the occurrence and production of fire clays and diaspore clays and lists of producers of clays and clay products are given. One analysis of diaspore clay is stated as follows: moisture = 0.60%; loss on ignition = 14.00%; silica (SiO_2) = 9.30%; alumina (Al_2O_3) = 73.73%; iron oxide (Fe_2O_3) = 0.57%; soda = 2.00%; potash (K_2O) = 0.52%. Brief statements concerning the occurrence and production of barytes, together with a directory of producers, are included.

E. D. ELSTON.

Refractory clay from Mattagami River. *Twenty-seventh Annual Report. Ontario Bur. of Mines,* Part 1 (1918).—Deposits of highly refractory, excellent, white fire clay have been discovered on Mattagami River opposite an island at the foot of the Long Portage. The material was tested by Mr. J. Keele of the Mines Department, Ottawa, and was pronounced by him to be one of the most refractory clays yet found in Canada. Furthermore, this clay is stated by Keele to be suitable for sanitary porcelain, vitrified floor tiles and wall tiles, and probably electrical porcelain. A red clay found on the same property proved to be semi-refractory in character and would probably make good fire brick when mixed with the white fire clay in the proportions of one part red clay with two parts of white clay. These deposits, however, are far from the railroad lines. E. D. ELSTON.

Refractories.

The Ceramic Society. *Pottery Gazette,* No. 503, 489–496 (1919).—At the monthly meeting of the English Ceramic Society, held April 14, the following papers were read:

"The Corrosive Action of Frits on Refractories," by R. L. Johnson. The method of making slagging tests by placing slag in holes, drilled in brick, is not satisfactory because the protective coating on the brick is destroyed by drilling. Using china rings on the surface of brick and putting frit in them was found to give more satisfactory results.

"The Substitution of Apatite for Bone Ash in China," by N. B. Davies. Tests were made to substitute Canadian apatite for bone ash. The following bodies were used:

	No. 1.	No. 2.	No. 3.	No. 4.
Apatite.....	42%	39%	35%	32%
Feldspar.....	15	16	17	19
China clay.....	33	34	34	35
Flint.....	10	11	14	14

All bodies had excellent casting and working properties. Greatest translucency was attained in body 4 at cone 9. The ware had a slight greenish tinge. In the discussion which followed it was stated that ware made from similar bodies lost their shape in burning.

"The Heat Conductivity of Porous Materials and Heat Insulation of Kilns," by Dr. J. W. Mellor. In calculations as to the rate of heat travel through a solid body and across adjacent air space with solids of different sizes, the author found that with cubes having a side of 0.1 cm., at 1400° C, the amount of heat carried per second across the air space and across the solid is the same. If pore space is less than above, the temperature in the air space would be higher than that of the adjacent solid. If, however, the pore space were 0.5 cm., the temperature would be much lower, coming out at just about enamel kiln heat, showing that the insulating properties of the air were breaking. With a pore 0.5 cm., above 730° C the air was a poorer insulator than was a solid. Certain experiments to verify the calculated results are being conducted and will later be published in the Transactions.

H. G. Schurecht.

Notes on refractory materials. By a Correspondent. *The Pottery Gazette*, No. 505, 727–730 (1919).—For resistance to action of flue dust, etc., the bricks are protected either by actual fusion of the surface of special brick or by painting with a mixture of carborundum plus 10% of fire clay or silicate of soda. The resistance to slag action is much greater when the brick are made under high pressure than when made with low pressure. Brick made of bauxite clays are giving good results in ports of gas-fired furnaces and in the crown of an electric furnace. A vitrified glass pot is not corroded by glass but a non-vitrified pot is strongly attacked above 1200° C and therefore it is suggested that empty pots be burned to 1350° C. In making silica brick it has been found that brick made of fine-grained quartzites, with a cement of amorphous silica, are more readily inverted into cristobalite or trydimite than those made from coarse-grained quartzite. H. G. Schurecht.

Glass.

The use of lime in the glass industry. F. Gelstharp. *Rock Products*, 22, 30 (No. 18, 1919).—Manufacturers of bottles and pressed and blown wares have always preferred to use quicklime, CaO, and some prefer hydrated lime, $Ca(OH)_2$. The general impression of such users is that the quicklime and hydrated lime give them better melting conditions and a glass of better color than can be made by use of limestone. In recent years some of these manufacturers have been induced to use limestone and have been quite successful in producing a glass at least equal to that formerly produced from quicklime. For window and plate glass limestone has always been used. When an attempt was made to use quicklime or hydrated lime, trouble in melting was experienced unless special care was exercised in the mixing with the other materials and in frequent testing of the lime. For plate glass, limestone should contain not over 1.5% MgO, although the content may be higher if the rock is uniform in composition and certain changes are made in the glass mix to control the degree of viscosity of the glass. Variation in the MgO content affects the melting conditions and the uniformity of the

glass. MgO compared to CaO in chem. equivs. produces a glass less fluid under like conditions, but compared by weight causes little difference in fusibility. MgO tends to prevent devitrification and lowers the coefficient of expansion. Limestone for glass should be ground so that 100% passes a 10- or 12-mesh screen and 90% passes 16-mesh, as this gives a degree of fineness that mixes well with the batch and gives a quick melt. The following are analyses of limestones from various localities and which have been found satisfactory for the several kinds of glass:

	SiO_2.	Fe_2O_3.	Al_2O_3.	$CaCO_3$.	$MgCO_3$.
1	1.00	0.13	0.05	97.81	1.01
2	0.23	0.10		98.12	1.55
3	0.60	0.05	0.08	98.56	0.71
4	0.50	0.14	0.04	89.07	10.25
5	0.84	0.06		98.60	0.50
6	0.44	0.26		98.50	0.80
7	0.94	0.10		98.05	0.91
8	0.08	0.02	0.12	99.25	0.53

The limestone must be as pure as possible, especially for the finer grades of glass. Al_2O_3 in the form of small amounts of calcareous shale will not give trouble, but in the form of ganister will give endless trouble by producing striae and stones. For plate glass, less than 0.10% Fe_2O_3 is desired and 0.15% is the maximum allowable. Dolomite limestone may be used in the glass batch. It is generally employed in the calcined form, and in tank furnaces where variations in the ratio of MgO to CaO can be more easily tolerated than where melting is done in pots as in the manufacture of plate glass. Chemical composition alone and not physical differences of the limestones affects the quality of the glass. F. A. KIRKPATRICK.

Enamels.

Acid test on enamels. W. D. COLLINS. *J. Ind. and Eng. Chem.*, **14**, 8 (1919).—Tests were made at the U. S. Bureau of Agriculture for acid resistance of 61 samples of enameled kitchen-ware from 26 American manufacturers. The principal test consisted of boiling 500 cc. 4 % acetic acid in the vessel for one-half hour. Some tests were also made with 1% tartaric and citric acids, the results of which corresponded to those obtained with 4% acetic acid. About half of the samples of white- and gray-ware suffered no loss of "glaze" on treatment with 4% acetic acid, while nearly all blue-ware was badly affected with 2% acetic acid. The amount of material dissolved in each case corresponded with the loss in "glaze." Seventeen samples from nine manufacturers gave no test for antimony. White and gray enamels contained about equal amounts of antimony according to tests, 34 out of 47 samples giving up 0.5 to 2.0 mg. of antimony when boiled with 4% acetic acid for 6 hours. Tests showed no antimony present in four blue enamels

tested. Lead was found in ware from only one manufacturer. Pieces of enamel chipped off several vessels under a heat treatment no more severe than might be received by any cooking vessel in use. R. R. DANIELSON.

Metallic coating for the rust-proofing of iron and steel. *Circular of the Bureau of Standards, No.* 80.—Attempts to protect iron and steel against corrosion have led to the use of various materials for coatings, such as metallic coatings, oxide coatings, organic coatings such as paints and varnishes and vitreous enamels. This circular deals particularly with metallic coatings but contains much material of interest to those coating metals with other substances, such as vitreous enamels. The section on pickling and cleaning metals and the effects of these processes on the mechanical properties of steel will be of special interest to manufacturers of enameled wares. A well-selected and classified bibliography dealing with the general subject of corrosion is appended. HOMER F. STALEY.

Cement and Lime.

The Ternary System CaO–MgO–SiO_2. J. B. FERGUSON and H. E. MERWIN. *Am. J. Sci.*, 48, 6 (Fourth Series) (1919).—The solidus-liquidus relations, the general behavior on heating to high temperatures and the properties of the compounds and solutions formed have now been determined for the four ternary systems CaO–Al_2O_3–SiO_2, CaO–MgO–Al_2O_3, MgO–Al_2O_3–SiO_2, CaO–MgO–SiO_2. These comprise the four possible ternary systems which can be formed from the oxides: CaO, MgO, Al_2O_3, SiO_2. The next step in this important series of investigations has been stated by the workers of the Geophysical Laboratory to be a study of the relations in the quaternary system CaO, MgO, Al_2O_3, SiO_2. In previous publications of the Geophysical Laboratory and of the American Ceramic Society applications of these results to ceramic problems such as the manufacture of Portland cement and of porcelain and other pottery wares have been made. The results should not, however, be used in these connections without the aid of data on the practical properties of the ceramic products. For example, the results of the above investigations indicate that in a certain type of porcelain, CaO would give a greater degree of fusion with like heat treatment than would MgO. From this it might be thought that CaO would give greater translucency than MgO. The reverse is true in this particular porcelain which is of the average American type. Other factors, possibly small amounts of other oxides, seem to govern the translucency more than does the supposedly greater fluxing action of the CaO. The relations found in the system CaO–MgO–SiO_2 are shown in Fig. 1. The wt. % compn. of any mixture of the three oxides is represented within the triangle. The proportion of any one oxide may be found by measuring the perpendicular distance from the point representing the mixture to the side opposite that representing the oxide, the total distance from the oxide apex to the opposite side being 100. For example, the % of SiO_2 in the mixture which happens to be represented by

the point designated $CaO.MgO.SiO_2$ may be read from the diagram as 38%. The areas bounded by the heavy full lines are called "fields" of stability of the oxide or compound designated for each. In the area or "field" designated as CaO, all mixtures when melted and then cooled slowly will crystallize out first CaO, then some other oxide or compound. In the area designated $2CaO.SiO_2$, this compound is the first to crystallize, and so on for all the areas. Some parts of the diagram representing certain kinds of mix-

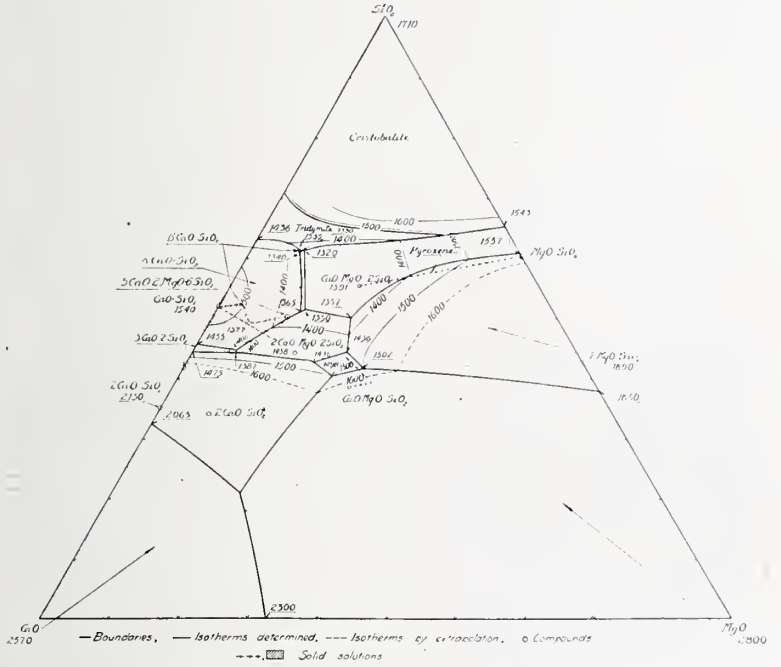

FIG. 1.—A concentration diagram upon which the isotherms showing the temperatures of complete melting of the various compositions are drawn. The diagram is on a wt. per cent basis.

tures and compounds are complicated by the fact of production of solid solutions, as for example, the pyroxenes which are mixtures of $CaO.MgO.2SiO_2$ and $MgO.SiO_2$. The system $CaO–MgO–SiO_2$ is the most complicated of the four systems in the series on account of the great number of solid solutions which may form. As indicated in the diagram, there are 14 crystalline phases (oxides, compounds, and solid solutions) found in this ternary system. There exist also 14 quintuple or invariant points at which three crystalline phases and a liquid can co-exist, and of these, six are true eutectics. These

quintuple points are the points of intersection of three boundary lines and when the temperature along each of the three lines falls as they approach the point of intersection the point is called a eutectic, the mixture having a lower temperature of complete crystallization than surrounding mixtures. The broken lines and the light full-lines in the diagram pass through points representing mixtures having like temperatures of complete melting. It is noted that a certain mixture represented in the upper left portion of the diagram and having a composition of 32% CaO, 7% MgO, 61% SiO_2 has a melting temperature of 1320° C, being the lowest melting eutectic mixture in the system. If the 7% MgO were left out of the mixture the melting temperature would be about 1500° C. It is such low melting mixtures as these which become effective as fluxes in pottery bodies and glazes. Comparisons are made between some of the compounds and solutions produced and those found in nature. In addition to 13 crystalline phases which are definite compounds there are described 5 series of solid solutions. Optical properties of these compounds and solutions were determined. While these investigations have been carried out mainly as studies of the formation of rocks and minerals in nature they are useful in studying the fundamental principles of the production of silicate products in the industries.

F. A. KIRKPATRICK.

Alkalies in cement clinker: Their relation to the setting time. B. F. ERDAHL. *Concrete, C. M. S.*, **15**, 17 (1919).—The nature of the alkalies in flue dust has been extensively studied, while little attention has been paid to the non-volatilized alkalies in the clinker. The remaining alkalies may have a great effect on the quality of the cement. This applies especially to plants using the Cottrell precipitation system for abating the dust nuisance and recovering the dust for clinker, potash, or for both purposes. Theoretical considerations indicate that the alkalies in the raw materials are entirely decomposed by the heat of the clinkering zone and the alkali vapors immediately recombined with the liberated SO_2 to form sulphuric compounds of higher volatilization temperatures than the original ones. SO_2 in the coal aids in the formation of sulphates. The amount of CO_2 present in the clinker is very small. Flue dust, clinker, and c. p. $CaCO_3$, each in ten g. amounts, were heated at 1050° C for 2 hrs., then at 1150° C for 30 mins. By considering the $CaCO_3$ determination as a blank, the dust was found to contain CO_2 equivalent to 0.06% K_2CO_3, and the clinker an amount equivalent to 0.03% K_2CO_3. It is possible that volatilized potash and soda recombine with the coal ash forming a silicate slag, which may remain undecomposed and come out with the clinker. However, there can hardly be any doubt that the greater part of the alkalies exist in the clinker as sulphates, some as plain K_2SO_4 and Na_2SO_4, and some perhaps as double salts of these, with lime or silica. In order to strengthen this belief, two representative clinkers were analyzed for water-soluble alkalies by boiling 12 hrs., and for total alkalies by the J. Lawrence Smith method. The results were as follows:

Sample No.	% total K_2O.	% water-soluble K_2O.	% total Na_2O.	% water-soluble Na_2O.
1	0.15	0.12	0.26	0.10
2	0.28	0.26	0.33	0.19

From this it is seen that the greater part of the alkalies in the clinker is composed of water-soluble compounds and that the K salts are more soluble than the Na salts. The most important effect of the alkalies in the cement is that of change in the time of set. Solutions of alkali sulphates, carbonates and silicates were prepared, ranging in strength from 1% to 5% of the anhydrous compounds. These solutions were added to a cement of good quality which had an initial set of 180 mins. and final set of 380 mins., by the Gilmore needle test. 22.5% of solution (by wt.) was added in all cases, the temperature of air and water was between 21° and 22° C and all pats were sound. The results are given in the following table:

Solution used.	% alkali by dry wt	Time of set. Minutes.	
		Initial.	Final.
Water	0	180	380
K_2SO_4	.225	150	300
	.675	135	260
	1.125	135	250
Na_2SO_4	.225	120	275
	.675	110	260
	1.125	90	230
K_2SiO_3	.225	150	370
	.675	125	310
	1.125	120	300
Na_2SiO_3	.225	160	335
	.675	125	300
	1.125	100	260
K_2CO_3	.225	65	260
	.450	11	150
	.675	7	20
	1.125	5	10
Na_2CO_3	.225	65	260
	.450	9	36
	.675	6	12
	1.125	5	12

The conclusions are as follows: (1) The alkalies exist in clinker and cement mainly as readily soluble K and Na sulphates, and as slowly water-soluble double sulphates of the alkalies and lime. (2) The smaller part of

the alkalies in clinker and cement exist as water-insoluble or very slightly soluble alkali slags (silicates), consisting chiefly of Na compounds. (3) Traces of K and Na carbonates may be present in clinker and cement, but hardly in amounts to affect their physical qualities. (4) The alkali sulphates and silicates have a somewhat quickening influence on the settling time of cement. The alkali sulphates are also detrimental to the free use of gypsum as a retarding agent, as a smaller amount must be added to keep within the specification of 2% SO_3. (5) The alkali carbonates are very dangerous to the setting qualities of cement but are not often found in large amounts. Alkalies do not have any determining effect on the soundness of cement.

F. A. KIRKPATRICK.

CERAMIC PATENTS.

G. E. MIDDLETON, *Abstractor.*

Process of molding lavatories. D. W. MCNEIL. U. S. 1,311,410, July 29, 1919. A removable core is used in molding the overflow passage in the back wall of the lavatory thus avoiding the necessity of applying the walls of this passage to the exterior of the bowl and affecting the adhesion by means of slip.

Process of separating mica from feldspar. G. J. BANCROFT. U. S. 1,310,939, July 22, 1919. Feldspar and mica are ground together and separated by washing, the feldspar being lighter than the mica.

Process of producing potassium hydrate from green sand. B. A. PEACOCK. U. S. 1,309,744, July 15, 1919. Glauconite or feldspar is heated for 15 or 30 minutes with KOH or K_2CO_3 to form a potassium zeolite which is very slightly soluble in water. To this zeolite, $Ca(OH)_2$ and water are added and the mixture boiled for an hour. The $Ca(OH)_2$ reacts with the small part of the zeolite which has gone into solution, precipitating a water-insoluble calcium zeolite and leaving KOH in solution.

Process of treating silicates containing potassium and aluminium. L. A. EBERHARDT, U. S. 1,310,413, July 22, 1919. Potassium and aluminium salts such as potash alum and aluminium sulphate are recovered from silicates containing potassium and aluminium. A finely divided mixture of sericite, feldspar or other potassium-aluminium silicates, fluorspar and gypsum, is calcined at a red heat in a rotary furnace for two hours. The calcined mixture is then treated with H_2SO_4 in sufficient amount to form the desired sulphates. The $K_2Al_2(SO_4)24H_2O$ and $Al_2(SO_4)_3$ are separated by evaporation.

Process of decomposing potassium silicates. S. R. SCHOLES. U. S. 1,312,053, August 5, 1919. Orthoclase feldspar, leucite, sericite, lepidolite, the micas or any other like potassium aluminium silicates may be treated and if only alumina and silica are the products sought, kaolin, sillimanite or

other aluminium silicates may be used. Equal parts of the silicate are mixed and fused with Na_2CO_3 or K_2CO_3. The resulting glass is pulverized, is then digested at about 160° C, corresponding to about 90 pounds steam pressure, for about two hours. It is then filtered and washed until free from alkalies. The washed precipitate consists of a compound containing alumina. The filtrate is treated with CO_2, the alkali metals being converted into carbonates from which the silica separates as a colloidal precipitate.

Process of decomposing natural silicates. B. A. Peacock. U. S. 1,310,770, July 22, 1919. Potassium-bearing silicates are decomposed with the aid of H_2SO_4. Feldspar, leucite, glauconite, etc., are mixed in finely divided condition with a magnesium-bearing silicate, such as serpentine or dunite, and sufficient H_2SO_4 added to convert substantially all the potassium and magnesium present into sulphates. This pasty mass is placed in a heavy masonry receptable 10–25 feet in depth called a "den," similar to a silo. The serpentine and dunite are attacked by the acid in the cold, the temperature of the mass being elevated to 150° C and the pressure in the den rising. The potassium-bearing silicates are then decomposed. After 24 to 48 hours a double sulphate of potassium and magnesium may be recovered. It is treated with $CaCl_2$ to form a double chloride and this is digested with slaked dolomite resulting in $CaCl_2$ and KCl in solution and precipitation of all magnesium as hydroxide. The KCl is obtained by evaporation.

Process of purifying aluminous materials. O. Hutchins. 1,310,342, July 15, 1919. This process relates to the production of an aluminous abrasive material of relative high purity, containing not over 1% each of Fe_2O_3, TiO_2 and SiO_2, and consists in fusing aluminous materials such as bauxite, in the presence of carbon insufficient in amount to reduce all the Fe_2O_3, TiO_2 and SiO_2 contained therein, separating the aluminous product from the reduced impurities and re-fusing the product in the presence of sufficient carbon to cause a further reduction of the above oxides.

Abrasive Wheel. H. R. Power. U. S. 1,310,291, July 15, 1919. The voids of an abrasive wheel having a ceramic bond are filled with polymerized benzofuran or paracoumarone. This substance has a relatively high melting point, and a grinding temperature of 90° C may be reached before it begins to flow.

U. S. 1,310,292 discloses the use of a mixture of paraffin wax and paracoumarone for impregnating porous abrasive wheels having a ceramic bond.

Abrasive Wheel. H. C. Martin. U. S. 1,310,360, July 15, 1919. An abrasive wheel having a silicate bond the voids of which are filled with a chlorin derivative of naphthalene such as Halowax, $C_{10}H_4C_{14}$.

Lining for acid pots. C. F. Curtis. U. S. 1,311,051, July 22, 1919. An acid-resisting cement is said to be made from the following ingredients: sawdust 30%, portland cement 30%, whiting 10%, waste residue of pyrites furnaces 30%.

Flowing molten glass. J. Whittemore. U. S. 1,310,225, July 15, 1919. This invention relates to the method of segregating molten glass into individual mold charges or gobs in which the glass is periodically severed by a mechanical shear as it is discharged vertically downward from the furnace in the form of a column. When a gob has been severed the cutter is immediately withdrawn and the glass exudes in the form of a heavy stream. At the same time a heating cup swings beneath the flow opening and up into engagement with the bottom plate of the furnace. This cup is provided with a refractory lining heated by an electric coil, the heat serving to reheat the chilled, cut end of the glass and eliminate the shear marks. The entire surface of the glass column is heated. The glass is prevented from actual contact with the walls of the cup by means of heated compressed air introduced through perforations in the cup. After sufficient heating the cup is swung downward and to one side and the gob is sheared and dropped into the mold.

Method and apparatus for manufacturing window-glass cylinders. Campbell. U. S. 1,312,305, August 5, 1919. Variations in the surface tension of glass in the pot cause the cylinder to move away from the center and into contact with the side of the pot. This may be avoided by leveling and unleveling the pot either before or during the draw which regulates the cooling and equalizes the temperature of the glass in the different parts of the pot.

Glass apparatus. C. Donaldson, J. J. Bloxam and J. K. Brooks. U. S. 1,311,804, July 29, 1919. A support for glass cylinders after drawing and during the capping operation. This support or horse is provided with a reheater for the bait which is rendered automatically active when the bait is placed upon it.

Glass-working apparatus and process. F. L. O. Wadsworth. U. S. 1,311,474, July 29, 1919. When molten glass is allowed to flow downward from a melting furnace to a mold it becomes externally chilled. If, when the flowing column of glass is cut, it is cut too near the discharge orifice, the chilled walls and the end chilled by the cutting prevent the continued free flowing of the glass and cause delay. If, on the other hand, the column is cut too far from the discharge orifice the result is an immediate resumption of the flow before the second mold is in readiness. This is regulated by the use of artificial chilling means employing as cooling media steam or compressed air.

Method of and apparatus for gathering glass by suction. E. Roirant. U. S. 1,311,249, July 29, 1919. Ordinarily in gathering glass by suction the lower portion of the gathering means causes a cooling of the glass, by reason of the metallic contact of the device with the glass, with the result that after several successive gathering operations at the same place the glass can no longer be used. This invention is said to obviate this difficulty. A float of

silica bricks rests upon the surface of the molten glass. The suction gathering means is brought with its lower end in contact with a cavity in the float at the same time slightly submerging the latter and causing the glass to rise in the lower part of the gathering means. Gathering is then effected by the use of suction.

Combined glass machine. G. A. Shields. U. S. 1,310,451, July 22, 1919. Plate or sheet glass is flattened and scored while in molten condition as it emerges from the delivery or discharge end of the tank-furnace and the thickness of the glass is determined by an adjustable gate governing the discharge of the molten glass. The transparency of the glass is regulated by varying the vertical drop from the furnace, the longer the drop, the more transparent the glass.

Glass drawing apparatus. H. K. Hitchcock. U. S. 1,309,942, July 15, 1919. This apparatus is designed particularly for drawing glass cylinders. The motive power for drawing the cylinder is also utilized in swinging the take-down frame; at the end of the draw the power is automatically shifted from the drawing mechanism to the take-down apparatus. The air supply is taken care of in a simple and effective manner and provision is made for guiding and steadying the drawing mechanism.

Apparatus for handling glassware. R. LaFrance. U. S. 1,310,194, July 15, 1919. A switching device receives bottles as they are delivered from a glass-forming machine and directs them alternately to setting-up devices by which they are placed in an upright position to be conveyed to an annealing oven or lehr. It is said that single setting-up devices have been found incapable of sufficient speed to enable them to be used with some automatic bottle machines. The present invention avoids this difficulty by providing two setting-up devices in combination with the oscillating switch.

Horse for glass cylinders. J. H. Campbell. U. S. 1,312,306, August 5, 1919. The several saddles of this horse are so arranged that pressure on one is transmitted to the others, thus evenly distributing the weight of the cylinder. This is accomplished by mounting the shanks of the saddles upon a common balanced lever, fulcrumed and spring-supported at its center.

Kiln. F. Munhollon. U. S. 1,311,286, July 29, 1919. The object of this invention is to distribute evenly and thoroughly utilize the heat in a down-draft kiln. Alternating with the bag-walls along the inner wall of the kiln are a series of vertical flues open at both ends and affording a connection between the space beneath the checker floor and the interior of the kiln adjacent to the crown. In each of these flues is a vertically directed nozzle designed to inject compressed air or steam thereinto. The pressure of the fluid used is sufficient to effect an aspirator action with the result that hot products of combustion which have passed down through the checker floor are whirled upward through the vertical flue and debouched against the crown of the kiln.

Enameling-kiln. G. H. BENJAMIN. U. S. 1,311,487, July 29, 1919. The bath tubs or other ware to be enameled are placed upon trucks and passed successively through a preliminary heating chamber, a coating or enameling chamber and a secondary heating chamber, the temperature gradually increasing in the preliminary heating chamber and gradually decreasing in the secondary. During the enameling proper, heavy doors shut off all communication between the coating and heating chambers so that workmen may enter and sprinkle on the enamel without any considerable physical discomfort.

Process for burning brick. F. B. LAMBERT. U. S. 1,311,978, August 5, 1919. The kiln used in this process is a temporary clamp or scove kiln. The noteworthy feature of the process is the so-called double use of heat. The firing is started using steam and oil burners, the supply of fuel being gradually increased until the arches are filled with flame from end to end. Then follows a period of intense firing resulting in an isothermal zone of incandescence extending throughout the kiln and just above the arches. When the proper heat condition has been attained as indicated by the settle, the firing is stopped and blasts of steam shot into the arches. The result of this treatment is to raise the isothermal zone of incandescence slowly up through the unburned brick. After twenty-four hours the steam is discontinued, and the natural draft allowed to complete the upward movement of the hot zone. Excellent results are said to have been accomplished by this method in the burning of Chicago hard-pan bricks.

Gas burner for kilns. R. H. MCELROY. U. S. 1,311,132, July 22, 1919. This burner consists of an elongated rectangular shell the inner end of which has a downwardly and forwardly slanting air passage which receives air through the top of the burner. Below the air passage there is a gas passage which receives gas through the bottom of the burner. This burner is said to give a long flame, obviating damage to the kiln due to localization of the heat.

Brick-machine. A. MALINOVSKY. U. S. 1,310,953, July 22, 1919. A dry-press brick-machine in which the end and edge faces of the bricks are stippled by means of stipple plates acting synchronously with the pressing rams.

Concrete composition. A. A. BENNETT. U. S. 1,310,520, July 22, 1919. A hard, strong concrete impermeable to water is said to be made from the following: Portland cement 1 part, aggregate consisting of conglomerate copper stamp sand 2 parts. The conglomerate runs from 1/4" in size to that which will pass through a screen of 200 wires to the inch and its composition is: SiO_2 60%, Al_2O_3 16%, Fe_2O_3 10%, CaO 3.5%, MgO 1.6%, Na_2O and K_2O 6%, metallic copper 0.6%.

Note.—Copies of these patents may be obtained for five cents each by addressing the "Commissioner of Patents, Washington, D. C."

ACTIVITIES OF THE SOCIETY.

Meeting of the Refractories Division.

Niagara Falls, N. Y., August 4, 1919.

The meeting was called to order by the Chairman immediately after the dinner given by the Society. There were eighteen persons present.

Attention was called to the necessity of securing papers for the next annual meeting. Upon canvassing the audience, Messrs. Harvey, McDowell Babcock, Howe, Klein, Shaw and Ross agreed to submit contributions.

The urgency of the situation with reference to the refractories used in the glass industry was then discussed. A motion to create a sub-committee on Glass Refractories was adopted. This Committee is to be instructed to coöperate with the Glass Division in every way possible.

A general discussion took place on the subject of research topics having a direct economic bearing. Among the problems brought out were the following: Sagger compositions; thermal expansion; effect of weathering; microstructure; critical spalling temperatures; kiln design; thermal conductivity; relation between size of specimen and spalling effect, dolomite brick, cements, coke oven insulation, and steam-pressed vertical lime-kiln brick. As a result of this discussion, motions to create two additional sub-committees, one on sagger compositions and one on microstructure, were seconded and carried.

It was finally voted that a monthly letter be issued by the Committee to its membership, keeping it informed concerning matters of importance and interest. The fund allotted by the Society to the Division is to be used for this purpose at first. Later it might be possible to enlarge the monthly letter to a printed pamphlet, supported by regular contributions from the membership of the Division.

RAYMOND M. HOWE,
Secretary.

AMERICAN CERAMIC SOCIETY.

Acquisition of New Members during September, 1919.

Associate.

Berkey, Paul L., Secretary-Treasurer and Plant Superintendent, Lava Crucible Company, Pittsburgh, Pa.

Chamberlain, C. H., Taylor Instrument Companies, Rochester, N. Y.

Dixon, Henry L., President and General Manager, H. L. Dixon Company, Pittsburgh, Pa.

Gleason, Marshall W., President and General Manager, Gleason-Tiebout Glass Company, Brooklyn, N. Y.

Jewett, Fred E., General Superintendent, Ball Brothers Glass Manufacturing Company, Muncie, Ind.

Lawton, Lewis H., Secretary and General Manager, Jonathan Bartley Crucible Company, Trenton, N. J.

Lippert, Charles G., Assistant Factory Superintendent, Schram Glass Company, Hillsboro, Illinois.

Moss, Lester M., Superintendent, Harrison Bulb Works of General Electric Company, Harrison, N. J.

Navias, Louis, Chemist, Bausch & Lomb Optical Company, Rochester, N. Y.

Pritchard, Geo. A., Vice-President, Buckman & Pritchard, Inc., 165 Broadway, New York City.

Rogers, Jas. B., Experimental Engineer, 6231 St. Lawrence Ave., Chicago, Illinois.

Rupp, Edwin M., 714 Lincoln Avenue, Middletown, Ohio.

Scott, David H., Ball Brothers Glass Manufacturing Company, Muncie, Ind.

Solon, Leon V., Art Director, American Encaustic Tiling Company, 454 Riverside Drive, New York City.

Wherry, Jas. B., Research Chemist, Refinite Company, Omaha, Neb.

Corporation.

H. L. Dixon Company, Pittsburgh, Pa.

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¶ Actual records were kept for 365 days of the burning of 9-inch, high-grade, refractory brick in one continuous tunnel kiln and in seven 30-foot round kilns, during which time 5,110,000 brick were burned in the tunnel kiln as against 5,040,000 in the seven round kilns.

¶ But this isn't all—the actual labor saving amounted to $5,808.00. Taking into consideration the necessary items of depreciation, interest on plant, maintenance and repairs, the average yearly cost for burning 1,000 brick in the continuous tunnel kiln system was $2.95 as against $6.20 in the round kilns.

¶ If you are really interested in the greater efficiency of burning, you will let us tell you more about the actual accomplishments of

The Didier-March Continuous Railroad Tunnel Kiln

Didier-March Company

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Contractors *Manufacturers of Refractories* *Engineers*

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Second:—This twin tunnel kiln allows a greater utilization of the fuel than a single tunnel kiln, as the heat from cooling ware is used for water-smoking the incoming ware.

Third:—This kiln allows an absolute and positive control of the firing zone, and in the firing zone a reducing or oxidizing condition can be maintained at will.

Fourth:—The cooling of the ware as well as the water-smoking and pre-heating in this kiln are absolutely automatic. It is impossible to get the receiving end of the tunnel too hot.

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In order to increase the stock of certain volumes of the Transactions of the American Ceramic Society, the Secretary of the Society has been authorized by the Board of Trustees to purchase a limited number of copies of Volumes II, V, IX, X, XII, and XIX, at five dollars each. Members or former members who are willing to dispose of their copies please communicate with

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Secretary, American Ceramic Society
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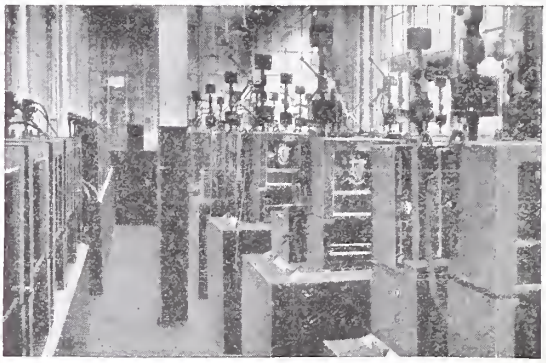

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May	6 1912..	4
Nov.	20 1912..	12
Feb.	11 1914..	1
Apr.	13 1915..	3
July	17 1916..	1
Dec.	7 1916..	1
Mar.	31 1917..	1
May	19 1917..	4
June	23 1917..	1
Oct.	26 1917..	3
Nov.	1 1917..	4
June	29 1918..	9

Beth. Steel Co.

June	16 1913..	1
Oct.	15 1913..	1
Nov	1 1913..	1
May	8 1914..	1
May	14 1914..	3
May	6 1915..	1
Dec.	14 1915..	2
Nov.	7 1916..	3
Jan.	4 1917..	1

Beth. Steel Co.

May	14 1917..	1
Aug.	14 1917..	1
Dec.	3 1917..	52
May	24 1918..	1
July	15 1918..	2
April	9 1918..	2
May	21 1918..	2
July	20 1918..	1
Jan.	22 1918..	5
Sept.	10 1918..	1
Oct.	4 1918..	1

Halcomb Steel Co.

Mar.	13 1916..	1
Oct.	27 1916..	1
Oct.	29 1918..	2
Dec.	27 1918..	1

Watertown Arsn'l

Sept.	23 1912..	1
June	30 1914..	1
July	1 1914..	1
Mar.	24 1916..	1
May	8 1917..	1

Watertown Arsn'l

June	4 1917..	1
June	25 1917..	1
Oct.	15 1917..	5
Oct.	23 1917..	18
Oct.	27 1917..	6
Jan.	5 1918..	1
Dec.	2 1918..	1

Packard Motor Car Co.

July	9 1908..	4
May	13 1914..	1
June	14 1914..	1
June	26 1915..	1
June	28 1915..	7
Aug.	16 1915..	1
Feb.	25 1916..	4
Feb.	3 1917..	1
Dec.	1 1917..	2
Jan.	11 1917..	14
Mar.	9 1917..	1
Dec.	16 1918..	4
April	22 1919..	4

DuPont Co.

Aug.	23 1915..	5
Oct.	15 1915..	2
Oct.	27 1916..	1
Oct.	28 1916..	1
Oct.	29 1916..	1
Jan.	5 1916..	1
Jan.	8 1916..	2
Jan.	8 1917..	1
Mar.	25 1917..	1
July	24 1917..	1
July	26 1917..	2
Nov.	11 1917..	1
Feb.	19 1918..	1
Mar.	28 1918..	1
Oct.	12 1918..	1
April	4 1919..	1

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May	11 1916..	1
June	26 1916..	10
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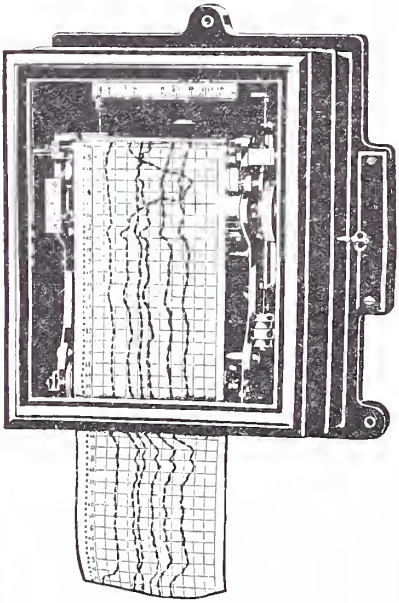

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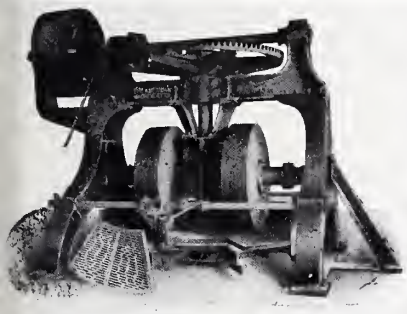

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JOURNAL
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AMERICAN CERAMIC SOCIETY

A monthly journal devoted to the arts and sciences related to the silicate industries.

Vol. 2 **November, 1919** **No. 11**

EDITORIALS.

SOME ASPECTS OF SCIENTIFIC RESEARCH IN RELATION TO THE GLASS INDUSTRY.[1]

By Edward W. Washburn,
Professor of Ceramic Chemistry, University of Illinois.

Although one of the most ancient of the chemical industries, the manufacture of glass, like most of the other ceramic industries, has been one of the last to feel the influence of modern science. This is, of course, due largely to the fact that the industry, being older than the science, had necessarily to develop along purely empirical lines. The rule of thumb methods, and their accompanying "trade secrets," which characterize the development of a complex industry by this method naturally bred a conservatism of view-point which did not easily appreciate the value of scientific research or the advantages of scientific control over the materials and the processes of the industry. Furthermore, it must be admitted that until comparatively recent times the chemistry and physics of high temperatures, such as those prevailing in the most important processes of this industry, were so little developed that the science was scarcely itself in a position to render important service to the industry, such a service for example, as organic chemistry has rendered to the synthetic dye industry which is a typical example of a great industry built upon a highly

[1] Address before the American Ceramic Society meeting held at the Fifth National Exposition of Chemical Industries, Chicago, Ill., September 24, 1919.

developed scientific foundation and completely under the control of scientific methods and the results of continuous research.

During the last quarter of a century, however, there has been a gradual change in this situation with respect to the glass industry. With the development of methods of producing, controlling and measuring high temperatures in the laboratory our knowledge of the chemistry and physics of high-temperature processes has gradually and steadily increased. With the expansion of our knowledge in this field have naturally come opportunities to apply its methods, laws and data to the solution of industrial problems. The more enterprising glass manufacturers began to employ chemists in their plants and in laboratories which they installed. Starting initially as a works laboratory, sometimes illy equipped and miserably housed, they busied themselves at first almost entirely with control problems and works' troubles. After they began to demonstrate their usefulness, their opportunities, responsibilities and resources both in men and equipment naturally increased and the beginnings of real research laboratories in connection with the glass industry appeared.

Then came the great war and America awoke to the realization that she must rely solely upon her own resources for the manufacture of all the different kinds of glass which she required. One of the first results of this situation was the quantity production of a grade of chemical glassware, the equal and in some respects the superior of that for which we had previously been dependent upon Germany. This new American glass is essentially a product of scientific research and was developed by men trained in the principles of modern chemistry and physics and having the necessary laboratory facilities at their disposal.

Then came America's entrance into the war and with it the realization that we should need hundreds of tons and numerous varieties of optical glass, that is, glass conforming to the most rigid specifications, as to color, transparency, homogeneity, and freedom from striations, bubbles, stones or other imperfections which might interfere with vision; and on top of these qualifications each of the various types required had to have a definite and rigorously fixed index of refraction and coefficient of constringence. The extent to which the glass in an officer's binoculars,

or the glass in a gunner's range-finder, or the glass in a submarine or trench periscope, met or failed to meet these rigid specifications would probably be measured in terms of human lives. The situation which existed in this country with respect to optical glass, at the time America entered the war, was thus a real crisis. It is not my purpose to tell you the story of how this crisis was met. This will be ably done by others on today's program who are competent to speak from first-hand knowledge of the problems met and solved. Suffice to say that the crisis *was* met and the problems which *had to be solved were solved* and their solution brought about a degree of coöperation between the practical man of the factory and the scientist of the laboratory which would probably have required many years to bring about without the stimulus of war. With this coöperation there naturally developed a mutual respect, understanding and appreciation on both sides which should mean much for the future of the glass industry in this country.

If today we put to the glass industry of America the question, "Do you know how to carry out the multifold operations necessary in order to produce good chemical-glassware, good optical glass, good plate glass, good X-ray bulbs, good incandescent bulbs, etc.?" the industry can truthfully answer, "Yes." Also, if we put the question, "Do you know how to do it efficiently?" the industry can still in many respects truthfully answer, "Yes," although in some cases, perhaps, in a rather weak voice. But if we put the question, "Do you know why you do thus and so, and do you know what happens at this or that stage of the process?" the industry must in the majority of cases reply, "We do not know."

Now, it is not enough to know *how*, we must also know *why*. For quality of product, it is perhaps sufficient merely to know how, but for efficiency, safety and confidence as well as for progress it is also necessary to know why. No industry which merely knows *how* is in a healthy condition. The feeling of confidence which comes with knowing the why of every step in the manufacturing process carries with it certainty of control, progressive lowering of costs, enhanced protection of the workmen, progress in the development of new and improved products, and a degree of insurance against such evils as might otherwise

attend the exhaustion or temporary scarcity of certain raw materials, fuels, machinery or other necessary supplies.

In the face of the imperative demands of war, or of the almost unlimited markets created by the destruction wrought by war, the necessity of operating with the greatest efficiency is not felt by an industry as it is in normal times with close competition and a market whose further expansion is dependent primarily upon lower selling prices. This is well illustrated in the case of the first two glass factories to resume production after the evacuation of Belgium by the Germans. These two factories, which I had the opportunity to visit last summer, are located in the same district and, therefore, subject to the same conditions regarding fuel, common labor and costs of materials. They are both engaged in the manufacture of window glass, for which, for some time to come, there will be an enormous demand for reconstruction in the devastated regions. In the first factory the glass is manufactured by the older process in which the "metal" is gathered directly from the furnace on the end of the blower's pipe and then blown into a long cylinder which is cut lengthwise, transferred to the flattening furnace and there flattened into a sheet by a second workman. In this factory the blowers are paid from $250.00 to $300.00 per month. In the second factory, operating under the Fourcault patents, the process is entirely mechanical, no labor, other than that employed in firing, being employed between the point at which the "batch" is shoveled into the tank furnace and the point at which the completed panes of glass issue from the furnace. The highest-paid workman in this plant received about 18 cents per hour. In spite of the apparently great difference in efficiency and in costs of the two methods of operation they were nevertheless both able to operate at a profit in a market where demand and not manufacturing costs is the principal element in determining the price of the product.

In the laboratories associated with an industry the work to be done may perhaps be roughly classified under three headings: First, *routine testing* of raw materials and products and similar work having to do with the control of the operation of the factory. Second, *works problems*, that is the solution of problems whose immediate purpose is the cure of some source of trouble in the

works or the improvement of some part of the process or product. Third, *fundamental research,* that is, research whose primary purpose is to find out the why of some stage of the operation, or to secure quantitative scientific data concerning the materials used, the processes occurring and the product obtained both in its completed state and in different stages of its manufacture.

It is the importance of the last type of work which I wish to emphasize particularly for it is only by the solution of these more general and fundamental problems that the scientific foundation of an industry can be laid. In the glass industry almost everything still remains to be done in this respect. For example, we know that the relation between the viscosity of the "metal" on the one hand and its temperature and composition on the other is one of the important elements which is associated with the operations, of stirring. gathering, drawing and blowing and yet one searches the literature in vain for any measurements of the viscosity of industrial glasses. The Germans may have secured these data, but if so, they have not published them. During the last two years we have been working on this problem at Illinois and have succeeded in perfecting an apparatus and in determining the viscosity-temperature curves for seven of the more important types of optical glasses.

Similarly, we know in a general way the mathematical theory of the process of fining and know that it is closely connected with the viscosity, with the surface tension and with the vapor pressure of the molten glass, but no measurements of surface tension of industrial glass seem ever to have been made, neither are any data concerning the vapor pressures of glasses at different temperatures or concerning the composition of the vapor available. Not even such a simple property as density seems ever to have been determined in the case of molten industrial glasses. Our knowledge of the reactions which take place in the melt, of the compounds which form, particularly those which crystallize out, and of the conditions under which they crystallize out, is still very fragmentary. Again, we know that soda can be substituted for potash to a large extent in certain optical glasses without impairing the index of refraction, but it is stated that this substitution is impracticable, because of the increased color which ac-

companies it. We do not know, however, the cause of this increased color, or whether it even is actually and primarily a necessary accompaniment of the use of soda.

There are numerous other examples of troubles for which the cure has been found, but concerning which we know nothing whatever of the true nature of the disease, or why the remedy employed resulted in a cure. It would not be at all surprising to find in some of these instances that the cure was not directly produced by the remedy employed, but resulted from some unrealized variable whose alteration accidentally accompanied the application of the remedy, the supposed remedy itself having nothing at all to do with the matter.

We know scarcely anything concerning the nature of the gases which remain in solution in the glass even after its completion nor do we know what rôle, if any, they play in determining the properties of the glass. It is quite reasonable, for example, to suppose that a method of manufacture which controlled the nature and quantity of these dissolved gases might produce an incandescent lamp bulb having a much longer life than those at present employed, and might place in the hands of the physicist a type of apparatus glass which would be of great value in high-vacuum investigations. The present methods of manufacture, as you know, exert no control whatever over this component of the glass.

The relation between some of the optical properties and the compositions of glasses has been extensively studied, but in the case of many of the other physical properties our knowledge is almost zero or at best only fragmentary. Suppose we desire, for example, to manufacture the most suitable glass for sealing in the lead wires of an incandescent light bulb, what do we know about the laws which govern the relation between the electrical conductivity of a glass and its composition? Almost nothing. I have seen a large Mazda lamp bulb destroyed in less than an hour's use owing to the electrolysis which occurred between the two leading-in wires, resulting in the formation of a layer of bubbles along one wire and a consequent destruction of the vacuum.

The purely scientific questions which present themselves for solution are almost infinite in number:

1. Under what conditions does one get metallic copper and cuprous oxide in a glass and how can one distinguish between them?

2. In a chrome-aventurine glass is the second phase chromic oxide or a chromic silicate?

3. Why does fluoride give a white glass with no red by transmitted light?

4. What compound gives the pink color with manganese?

5. What gives the red color in chrome pink?

These are a few questions which have been recently brought to my attention by an authority on colloid chemistry.

Some of the lengthy discussions which one finds in the literature concerning some of the questions arising in connection with glass manufacture are after all rather futile ones. The famous case of arsenic is an example. Much printer's ink has been employed in discussions as to what happened to the arsenic after it was put into the batch, just what influence it had on the properties of the glass and why it had this influence. Did it remain in the glass as As_2O_3 or was it oxidized as As_2O_5? In the discussion of this latter point, for example, by two such authorities as Dr. Hovestadt and Dr. Rosenhain, we find one of them claiming that if an oxidizing material is employed in the batch the arsenic remains in the glass as an arsenate, while if no oxidizing agent is employed no oxidation of the arsenic can occur and it is all driven out in the vapor. The other believed that since arsenic acid is easily reduced by heat alone any arsenic remaining in the glass was present as As_2O_3, and since arsenic acid was a weak acid it could hardly remain in combination with the alkalies and, therefore, its presence in the glass would result in the formation of insoluble impurities and a consequent opalescence. Now all of this discussion concerning the rôle of arsenic and its amount or condition in the finished glass has been a needless one, in so far as the questions raised were worthy of solution, because the problem under discussion was one which the resources of chemistry have long been in a position to definitely solve, if any one had taken the trouble to carry out the necessary experiments as has in fact been recently done.

There is thus no lack of chemical and physical problems in

connection with the glass industry whose solution requires only the painstaking work of trained investigators. It is not enough, however, to merely point out the great need of fundamental research in this field; it is necessary that definite steps be taken to prosecute this research as vigorously as possible. Now there are three types of laboratories in which research of this nature might be carried out. First, the research laboratories of the industry itself. These laboratories while as yet few in number may be expected to grow both in number, equipment and personnel and a portion of their resources should be devoted to some of the more fundamental problems common to the industry as a whole, as a part of their contribution to the general advancement of those branches of the physics and chemistry underlying the industry. Second, the government laboratories and the laboratories of research foundations, such as the Geophysical Laboratory. These may be expected to continue their work along these lines and should receive the support of the industry in their efforts to extend their facilities for such work. Third, the laboratories of the ceramic departments of the universities. Now, although we have some half-dozen departments of ceramics in the country at the present time, these departments have grown up around and been fostered by the clay industries and have as a natural result confined most of their activities in the past to these branches of ceramics. There exists nowhere in the United States a single professorship of glass technology. The endowment of such professorships by the industry would do much toward stimulating research in this field in our universities and in attracting young men to the study of this subject. And when all is said, it is, after all, the scarcity of trained men which is at the present time the greatest impediment to the more vigorous prosecution of research in this field. The Board of Advisors of the Department of Ceramic Engineering at the University of Illinois appreciated this situation and at its meeting in 1915 voted to recommend to the trustees that such a professorship be established by the University. Before any action could be taken, however, the disruption consequent upon our entrance into the war made it necessary to postpone the consideration of any expansion in the work of the Department and at the present

time the financial situation at the University is such that any expansion in its activities is out of the question. The universities of the country with but few exceptions have been unable to make any general increase in the salaries of their teaching staffs to meet the increased cost of living during the past four or five years and until they are in a position to correct the present desperate condition in which they find themselves, owing to the continued resignation of teachers of technical subjects, any plans looking towards expansion of activities by the creation of additional professorships could scarcely be justified.

I believe that no one of the institutions at which a department of ceramics exists is financially able at the present time to expand its activities. In fact, most of them have had to contract their activities and to struggle along with a reduced staff and smaller resources than they had before the war. If the glass industry is desirous of securing the coöperation of university laboratories in training additional men for the industry, and in helping to solve some of the fundamental problems in this field, some way of providing the needed financial assistance must be found.

The industry cannot afford to have the source of supply of its future technical staffs dry up and that is just what is occurring. A steady stream of requests for technically trained men for the glass industry and the other ceramic industries comes to my desk throughout the year. More and more of these requests are for men with graduate training, that is, men trained to do research. These men are not being produced. Few, if any, of our Ceramic departments are at the present time in a position to properly train such men for the glass industry. Such training is the most expensive which a university gives. Adequate fellowship funds are needed to enable the men to complete the course of training required to produce a research worker. Where six years ago a fellowship of four or five hundred dollars would pay a student's living expenses, today double that amount is required.

In the past the industrial world was content to rely entirely upon the universities for the advancement of science, but today there are many signs that scientific leadership is passing from the hands of the university to the hands of the great industrial research laboratories and the privately endowed research founda-

tions. Scientific research is becoming more and more costly and the universities as organized at present can no longer compete on terms of equality with the efficiently organized and adequately financed industrial and endowed research laboratories.

Thus, although no longer entirely dependent upon the universities for scientific research, the industrial world is and must continue to be entirely dependent upon them for the training of its technical personnel, and a part of the responsibility for seeing that such training is provided must in the future be accepted by the industries. Although the Sherman law forbids the formation of combinations in restraint of trade, there is, I believe, nothing in that Act which forbids the formation of combinations within an industry for the purpose of uniting on a program for furthering research on fundamental problems and for aiding the universities of the country in the work of training the technical personnel required by the industry.

SUPERIOR REFRACTORIES.[1]

By Ross C. Purdy.

This is a plea to the American Ceramic Society to initiate directly, or through the agency of the National Research Council, active and thorough researches of our refractory problems by a Federal bureau. This recommendation is made in spite of a keen appreciation of the excellent work which has already been completed and at present being undertaken by the Federal Bureaus, the National Research Council, the American Ceramic Society, the Refractories Association, the American Society for Testing Materials and other organizations. Much has been done by our universities and much by industrial concerns. In the presentation of this plea there is no forgetting of the advances we have made through the efforts of these several agencies and there is no thought but that these several bureaus, societies, universities and industrial concerns should continue independent researches

[1] Address before the American Ceramic Society meeting held at the Fifth National Exposition of Chemical Industries, Chicago, Ill., September 24, 1919.

on refractories as has been their wont, and as they no doubt are continuing, for there are advantages in independent investigations by several organizations each with its peculiar purposes, viewpoints, facilities, etc. Indeed, we want more of this dispersion of interest and activity in researches on refractories. It would be well if each of the several bureaus, societies, colleges, etc., would see the great need there is for progressive and aggressive investigations of our refractory problems and each independently initiate and prosecute researches in this the one branch of Ceramics in which we are the most backward in meeting the industrial demands for a better product.

Notwithstanding this keen appreciation of the benefits derived from independent investigations and researches by several, we must recognize the advantages of unrestricted facilities, not alone in expensive and varied equipment to meet every desired requirement, but also in facilities for very accurate observations and to the obtaining of trustworthy constants such as can be obtained only with highly trained investigators and expensive apparatus. We must appreciate also facilities for broad observations, not alone in the development and manufacture of refractories, but also as to the industrial needs for refractories. It is very obvious that the most rapid and the most certain progress in the development of refractories can be had only by an elaborate and aggressive investigation, an investigation that is beyond the possibilities if not beyond the scope of independent organizations of small financial resources and limited interest.

Then, too, the benefits would not be alone to the manufacturers of refractories, indeed not as much to them as to the users, and to the national and world-wide welfare. It does not require much breadth of thought to realize how dependent is our present-day industrial progress on the development and manufacture of superior refractories, refractories that are superior for the several purposes to those now being used. Consider for a moment the saving of time and of labor and of metal that is effected by the closed electric furnace over the open-top crucible for brass melting. Electric furnaces today are melting less than one per cent of the brass used and yet they are saving a million dollars' worth of zinc metal by reducing its loss from 6 to 9 per cent down to

one per cent and less. In this one operation alone they are not only saving metal but also labor, and at the same time are making the operation more tolerable for the operators. Examples could be multiplied by reference to the many heat-treating operations where with development of new equipment and processes they are effecting economies and bettering operating conditions. But these benefits cannot be fully realized until more suitable refractories are developed. The rapid development and adoption of electric furnaces for a large number of purposes has given rise to an urgent appeal for better refractories. The development and rate of adoption of the electric furnace has been phenomenal but is greatly retarded by want of suitable refractories.

There is an urgent need today for an understanding of the qualities or properties that are wanted in each of the large number of industrial requirements. This is no small task. It involves accurate observations and study of possible variations in operation control in the place and under the conditions that refractories are used. It requires a knowledge of metallurgy and of ceramics and above all it requires an appreciation of the relation of costs to service. The most suitable refractory will at the best be a compromise, not alone because of the antagonistic properties that are required, but also because of the economics of service involved. An adequate and worth-while industrial survey and research of refractory requirements is so far beyond the possibility and scope of individual organizations, and the benefits derived are so broad, affecting the cost and pleasure of living of every citizen, that it should be made under the auspices of a Federal Bureau where the expense is borne by general taxation.

If a discussion of the political economics involved were needed, it could be shown that since our national welfare is more and more dependent on industries as compared to agriculture, and since the farm and the industries are today more mutually dependent than at any previous time, all citizens should share in meeting these costs. And if the economic considerations were carried farther, it would be plain that efficiency, relating to time and money expended as balanced against the value of results obtained, would require the employment of the broadest-trained investigators, the most accurate and efficient equipment, and the most liberal

opportunities, all of which are possible only under Federal auspices.

Then, too, there should be extensive research of material resources and manufacturing processes. This should parallel the industrial survey. The two should be closely associated and each be given liberal support. A Federal bureau would secure for this a far better coöperation of the independent organizations than could any other agency, a coöperation that is very essential.

A great deal of progress has been made in the last two decades, not alone in refractories themselves, but also in our appreciation of the relative value of the chemical and physical means of judging their suitability. We no longer hear of the mistaken and much over-emphasized importance of chemical analyses of clays. We have not, and probably never will, discard the chemical analysis as being without value. It has a value, for it does give information, but we certainly have less faith today in the empirical methods of Bischof, Seger and Ludwig for estimating the refractoriness of clay from the chemical analysis. We have learned, too, that clays having the same chemical analysis but differing in physical and mineral character may have different values for refractory purposes. Indeed, it is not uncommon that a freshly mined clay would have but small value for use in refractories but will be made an excellent clay by merely weathering. This is an instance of the importance of texture and structural strength, factors that are not disclosed by chemical analysis.

We are appreciating more and more that the success of a clay for refractories is as largely dependent upon the physical properties that make it adaptable for manufacturing processes, as it is on its chemical and mineral composition. Indeed, the fact is slowly being appreciated that behavior of a clay in the usual fusion test is dependent on physical properties as well as on its chemical composition. No method yet devised can be substituted for the direct fusion test.

Decided progress has certainly been made in knowledge of the method of study, and progress has also been made in knowledge of what is essential in refractories, but we have made but little in the actual production for specific purposes.

It is not the purpose here to elaborate upon the relative values of the several methods of studying refractory materials and wares

or how to produce desired properties in the finished ware. We are making fair progress in these.

What we need today is the development of refractories that will remain practically constant in their physical and chemical characteristics through the maximum heat treatment to which they will be subjected when in use. We recognize that when a clay is burned in a fire-brick kiln the melting together of its mineral constituents is but partly progressed and that further reactions and solution will take place when the clay is subjected to more severe heat treatment. We also recognize the importance of such factors as time, oxidation, etc., in the progress of clay fusion. It is well known that clays that appear equal in the fusion test will differ in load-carrying capacities when subjected to heat treatments much less severe than those required to cause them to deform in the fusion test, and we recognize this as one indication that suitability of a clay refractory is not wholly dependent upon its ultimate fusibility, but in fact is more dependent upon its rate of fusion.

From the evidence that is already before us it is apparent that, for the most severe heat requirements, the refractory must have had its physical and chemical properties developed to their maximum; far beyond the possibility of alteration in any heat treatment to which they will be subjected in use.

If fused bauxite, fused alumina, sintered magnesia, or silicon carbide would meet all the industrial high-temperature requirements, there would be no need for this plea for Federal research on refractories. There are needs which these materials will not satisfy and for which materials, such as the spinels, sillimanite, zirconia, etc., are better suited. We have heard much of the possibilities of zirconia as a refractory and have had several industrial trials of refractories made of zirconia, but we know little aside from the fact that we cannot yet produce a zirconia refractory that is economically possible. The same is true of sillimanite, a most excellent refractory of exceptionally low electrical conductivity.

The value of these fused refractory-oxides lies, not alone in their extreme refractoriness, but also in their constancy in volume, capacity to withstand sudden temperature change, resistance to chemical and slag reactions and ease with which a desired texture

can be produced. Refractory articles from these materials average much higher in desired or required properties than any of the partially fused or partially sintered refractories. No refractory will carry load at high temperatures and withstand the destructive abrasive, slagging and temperature changes as will fused bauxite, silicon carbide or sintered magnesia. These same superior values might be found with other materials which have been previously fused.

The great problem with these materials, however, is their production at a cost compatible with the service they will give. The solution of this problem is not easy and certainly deserves the serious attention of a Federal bureau with extensive capacity to conduct investigations in the laboratory and field.

The plea that is being presented is that the American Ceramic Society initiate and secure coöperation by the several other Societies in a request for aggressive researches by some Federal Bureau in the development and economic promotion of refractories. This will include a definition of the possibilities of clay refractories; an improvement in such as the silica and magnesite refractories; but such a research should especially develop and define the most economic methods of production and use of such superior refractories as can be made of zirconia, the spinels, sillimanite, the nitrides, etc. This is no small program. It will tax even a Federal Bureau, if executed with the vigor that the present urgent industrial demands fully warrant.

This plea is respectfully submitted to the Standing Committee on Research for their careful consideration.

Norton Co.,
Worcester, Mass.

ORIGINAL PAPERS AND DISCUSSIONS.

THE GEOLOGY OF THE CATSKILL PORTLAND-CEMENT REGION.

By Robert W. Jones.

The production of portland cement, in the Hudson valley, was placed on a commercial basis about 1900. Several attempts at commercial production had been made as early as 1875 but the results were not exactly encouraging and until the Becraft mountain section began to produce the output was not very large. The opening of the Becraft mountain section was soon followed by active production in the adjacent Catskill region.

The geology of the Becraft mountain regions has been very thoroughly studied.[1] Becraft mountain is a noted outlier of Siluric and Devonic formations. The economic value of these formations has been very seldom taken into account by the geologists who have investigated the region.

West of the Hudson river, in the Catskill region, the identical formation, as exposed at Becraft mountain, excites no interest among geologists in general. The geology of this region is supposed to be simple. The great economic features of this region and its formations have not been realized. It is due to a general lack of information, among certain industries, that we are stating in as brief a manner as possible, the general geologic conditions of a portion of this limestone belt. Our statements can only be used as a basis upon which the detailed geology of any particular section may be enlarged.

[1] Amadeus W. Grabau, "Stratiagraphy of Becraft Mountain, Columbia County, N. Y." N. Y. State Museum, *Bull.* **69**, 1030–1079 (1902).

Map No. 1—Black Lake Region.

In describing the geology of the Catskill region, from the viewpoint of the cement technologist, we have taken the liberty of dividing the regions in two divisions, which, for purpose of reference, we have designated Black Lake and Van Luven Lake, taking the names from two locally well-known bodies of water. These two sections, almost directly north and south of each other, are separated by a distance of approximately two miles. The intervening section is economically of no importance as regards this discussion. It is occupied by the valley of the Catskill creek, where it breaks through the limestone area. Of the two divisions the Van Luven Lake is the only producer having three plants, known locally as the Acme, Alsen and Alpha. The Van Luven Lake area is commercially and practically distinct from the Black Lake area. The supply of limestone in the Van Luven Lake area, under the present methods of mining, is limited. The supply of limestone in the Becraft region is decidedly limited. The Black Lake region, as shown in the accompanying map (No. 1), is the southern extremity of a very great body of limestone.

The region west of the Hudson river consists of roughly parallel valleys following fairly close to the general direction of the river. In the area under discussion, a maximum of eight hundred feet elevation is reached. It is roughly divided into three natural physical divisions; the lower, level clay flats having a maximum of two hundred feet elevation and with comparatively few rock outcrops; the limestone region with a rather abrupt eastern boundary, an abundance of rock outcrops, a rather shallow soil deposit, and many north and south flowing swampy streams; the higher range of semi-isolated sandstone and shale elevations with a very abrupt eastern face and a low rolling western exposure. The cement-products region lies in the first two divisions. The general geologic structure of the region consists of an eastern sharply folded limestone and sandstone belt gradually dipping under the almost level Devonic shales and sandstones to the west. To the east of the limestone belt is an area of very sharply folded shales and sandstones of Ordovicic age.

Clay is used as the source of silica at all of the plants. It is secured from the immediate neighborhood of the mills. The clay deposits are fairly uniform in composition and are abundantly

able to take care of any production of portland cement for many years. The limestones are somewhat restricted by variation in chemical composition.

In order to properly understand the geologic and economic conditions of this region, we refer to the following tabular arrangement of the various formations exposed. Under the unit division we have purposely left out the rock designation, as certain of the formations vary from the exposures at their typical locality.

System.	Group.	Unit.
Devonic	Erian	Hamilton Marcellus
	Ulsterian	Onondaga Schoharie
	Oriskanian	Esopus Oriskany Port Ewen
	Helderbergian	Becraft New Scotland Coeymans
Siluric	Cayugan	Manlius Rondout Cobleskill
Ordovicic	Canadian	Normanskill

The **Hamilton** brown, gray and black sandstones and shales occupy the western and northwestern portions of both the Black Lake and Van Luven Lake areas. They are economically of little importance. They are the highest in the geological scale of the formations represented in the two sections. The contact between the Marcellus shale and the Onondaga limestone is seldom exposed.

The **Onondaga** limestone is decidedly distinct from the other limestones of the Hudson valley as far as physical characteristics are concerned. It is massive, heavy bedded and with an abundance of typical fossils. It is slightly lower in lime and considerably higher in silica and alumina than the other limestones of this

region. Owing to its comparative great resistance to the weather it is prominently exposed along the outcrop. It breaks well along the bedding and along two sets of joint planes at right angles with each other. It has not been used, in the Hudson valley, for the manufacture of cement. It is usually found with a comparatively low dip towards the west. Chert is present in abundance.

Below the Onondaga limestone there is found a rather thin bed of brown-colored, banded and impure limestone. This is the equivalent of the Scholarie grit. It is high in iron, alumina and silica. In general appearance it somewhat resembles the Onondaga and for purposes of mapping and sections we have included it with the Onondaga. In its physical characteristics it gradually passes into the Onondaga limestone above and into the Esopus grit below.

The **Esopus** grit occupies a prominent part in the surface geology of this section. It has no economic value. It is usually difficult to determine the correct bedding of this formation owing to the great abundance of joint planes running in every conceivable direction. The rock is of a very even, dark gray color and fairly coarse grained. In the general topography of the region its exposures appear as smooth rounded elevations generally showing a scarcity of vegetation.

Directly below the Esopus is a body of dark gray-colored sandstones and shales accompanied by thin isolated beds of limestone. Owing to physical characteristics we have mapped this with the Esopus. It is distinguished from the Esopus, however, by the more distinct parallel bedding, by the finer grain and by the presence of fossils. This formation is, in most localities, covered by surface materials such as swamp deposits or under water. This is a portion of the formation which represents an extension of the Oriskany sandstone towards the east. Below this formation, and usually carrying in the higher layers several distinct bands of chert, is found the Port Ewen limestone, also an extension of the Oriskany formation. This formation closely resembles, in physical features, the New Scotland limestone and is often mistaken for that formation. The several distinct beds of the Port Ewen vary both vertically and horizontally in chemical composition. The percentage of silica is compara-

tively high; magnesia is present, ranging as high as 10 per cent. The thickness of the Port Ewen varies from twenty to thirty feet.

The **Becraft** limestone, the main source of lime for the manufacture of portland cement, in this section, is found below the Port Ewen with a thickness varying from thirty-five to forty-five feet. Physically it is rather sharply defined from the Port Ewen. It is found in massive beds sometimes accompanied by thin shale partings. These shale layers form a natural parting plane. Fig. 1 shows the quarry conditions where the shale parting planes

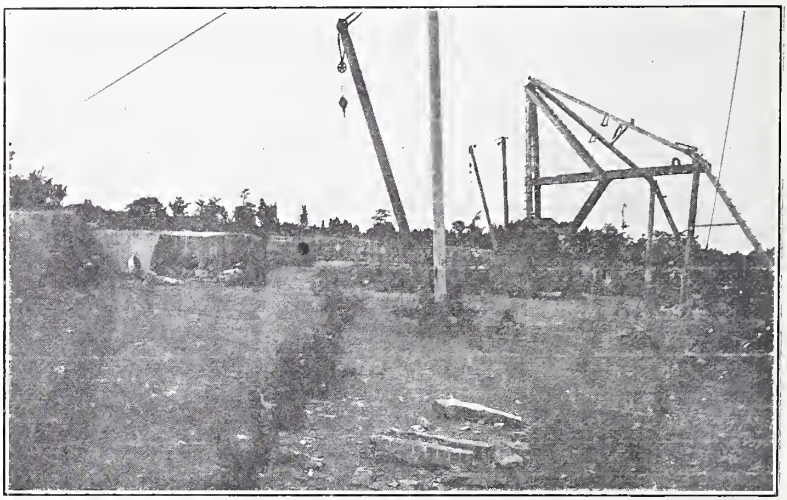


FIG. 1.

are prominently developed. A fresh exposure of the rock usually has a light gray color tinged with pink, the pink shade being derived from the iron-stained calcite of organic replacement. The Becraft limestone resists the action of the weather to a considerable extent and its exposures take a prominent place in the general topography.

The **New Scotland** limestone includes several distinct beds varying from a calcareous shale to a pure limestone. The shaly layers are apparently not suitable for the manufacture of port-

nd cement. They are high in alumina, iron and magnesia
id low in lime. Sulphur compounds are also somewhat abundant.
he basal layers of the New Scotland may be recognized by the
resence of several distinct layers of chert. Owing to the great
esemblance of the New Scotland and Port Ewen they are some-
mes mistaken for each other, but using the Esopus grit as a refer-
nce formation, there will be no difficulty in identification. The
ew Scotland limestone, comparatively speaking, does not re-
st the action of the weather and for this reason it is sometimes
fficult to locate the outcrops owing to the presence of abraded
weathered material. The limestone carries an abundance
fossils. An average thickness for the New Scotland may be
ken at seventy feet. Along with the Coeymans this formation
as been used as a source of cement material.

The **Coeymans** limestone is a rather massive dark gray rock
rather regular composition although in places it is compara-
vely high in magnesia. It is prominently exposed above the
anlius limestone and while it does not resist the action of the
eather, in regard to solution, as well as the Manlius, the absence
joint planes makes it a prominent capping over the rather
sily mechanically disintegrated Manlius. The Coeymans lime-
one varies in thickness with an average of about forty feet.
is easily identified from the New Scotland, using the chert
ds as a reference, and from the Manlius limestone by the rather
dden change in physical character of rock.

The **Manlius** limestone is found very prominently exposed
ong the base of the limestone ridge. Along the eastern border
the limestone ridge it is usually a member of the prominent,
ore or less exposed, syncline. The lower beds of the Manlius
e of a massive character and carry an abundance of typical
ssils. The middle and upper beds are comparatively poor in
ssil remains and vary physically from a thinly laminated charac-
r to an extremely massive columnar structure. Joint planes
e prominent to such an extent that the Manlius exposures are
sily picked out in the topography of the region. Material
om the laminated layers of the Manlius has a peculiar clear
inking sound when struck. The Manlius has a very irregular
ickness with a range of thirty to fifty feet. It varies greatly

in composition, especially with reference to silica and magnesia. This formation has been used as a source of lime for cement.

The **Rondout** limestone, known in the New York classification as the Rondout waterlime, is found directly below the Manlius. It has no economic value. It varies in thickness from several feet to a thickness where its admixture in the cement mixture would have to be taken into account. It may be distinguished from the Manlius by the brown color, due to weathering, and its massive character and lack of horizontal parting planes.

The **Cobleskill** limestone is a very thin bed of impure silicious limestone. It is economically in the same class as the Rondout and varies in composition from a pure limestone to a sandstone. It is not present as a continuous bed but thickens and thins from a few inches to several feet.

The **Normanskill** shales and sandstones, found usually to the east of the limestone belt, are in most cases covered by morainal deposits of clays. They are exposed prominently along the west bank of the Hudson river, in the bed of the Catskill creek, and at quite a few localities along the one hundred and two hundred foot contours, appearing as "islands" in the almost level clay deposits. They vary in composition especially with regard to alumina and silica. The shales are fairly high in alumina and will compare well with the clays as to silica. Usually they carry a lower percentage of lime than the clays. The sandstones, however, are rather low in alumina, high in silica, and usually carry a higher precentage of lime than the shales.

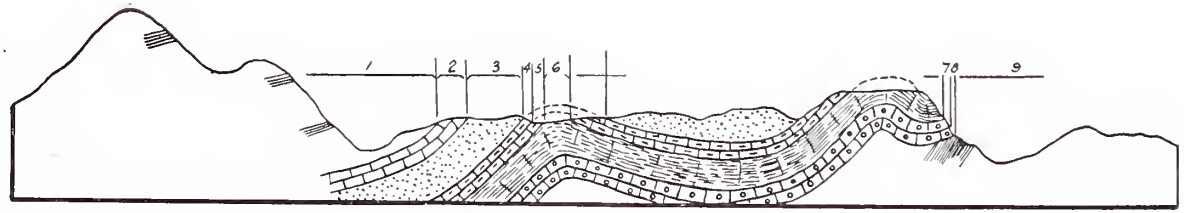
In the accompanying maps (Nos. 1 and 2) we have used a system of cross-hatching to designate the various formations. The classification is made with special attention to their physical characteristics. This classification, however, closely follows the usual palaeontological classifications. The vertical and horizontal cross-hatching represents, respectively, the Normanskill shales and sandstones and the combined Esopus grit and Oriskany. The diagonal cross-hatchings are used to designate the two limestone belts, the combined Onondaga and Schoharie at the west and the combined Cobleskill, Rondout, Manlius, Coeymans, New Scotland, Becraft and Port Ewen at the east.

Map No. 2—Van Luven Lake Region.

The reference points for the field examination of these deposits are the distinct Normanskill shales and sandstones at the base of the limestones, the Hamilton beds of brown, red and black shales and sandstones above the limestones, and the included mass of Esopus grit. The Onondaga, Port Ewen and New Scotland limestones have their chert layers and, therefore, we have a complete set of indicators for the location of the economic members. This statement is made on the assumption that the economic geologist will not be entirely familiar with the fossil remains and will, therefore, have to fall back on the physical characteristics.

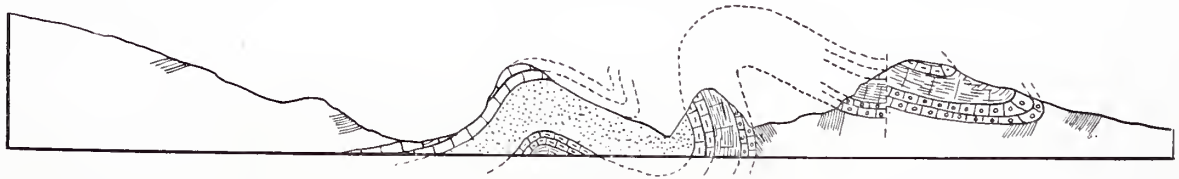
The commercial value of the productive beds has been more or less increased by the great amount of local folding and faulting. These beds have in many cases been brought to such a position that later erosion has placed them on the surface or very close to the surface. While the local folding and faulting is everywhere in evidence, at no place has it been extensive enough to throw any two physically distinct beds in contact. The character of the folds changes very suddenly along their strike. Each locality, to be properly understood, has to be studied independently and therefore the general scheme only of the folds is shown in the following sections (Sections 1 to 7).

The sections speak for themselves. The one persistent character that appears throughout the region and which is shown in these sections is the syncline along the eastern contact of the limestones. In the Van Luven Lake area this syncline is very sharp along sections 2, 3 and 4. This syncline gradually becomes less pronounced towards the south, with the exception that between sections 6 and 7 a series of very sharp local folds appear (Fig. 2) to again die out finally at the end of the limestone belt. Between this general syncline and the western contact of the limestone and the Normanskill deposits are usually a series of small simple folds. The greatest amount of displacement is along the eastern border. Between sections 6 and 7 the sharp folding and faulting has thrown into the productive area a considerable amount of waste material. This is well shown in Fig. 3 where the continuity of the productive layers is broken by a mass of slickensided, arenaceous limestone. Horizontal faults have in

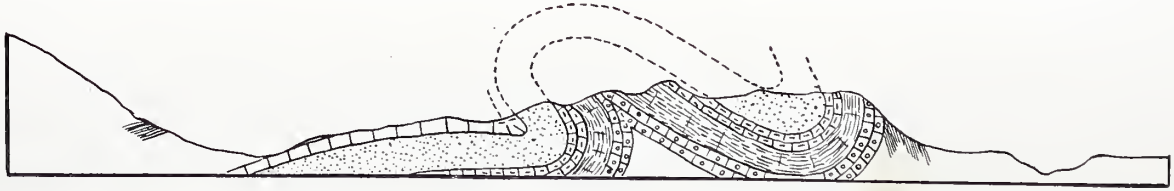


Section 1.

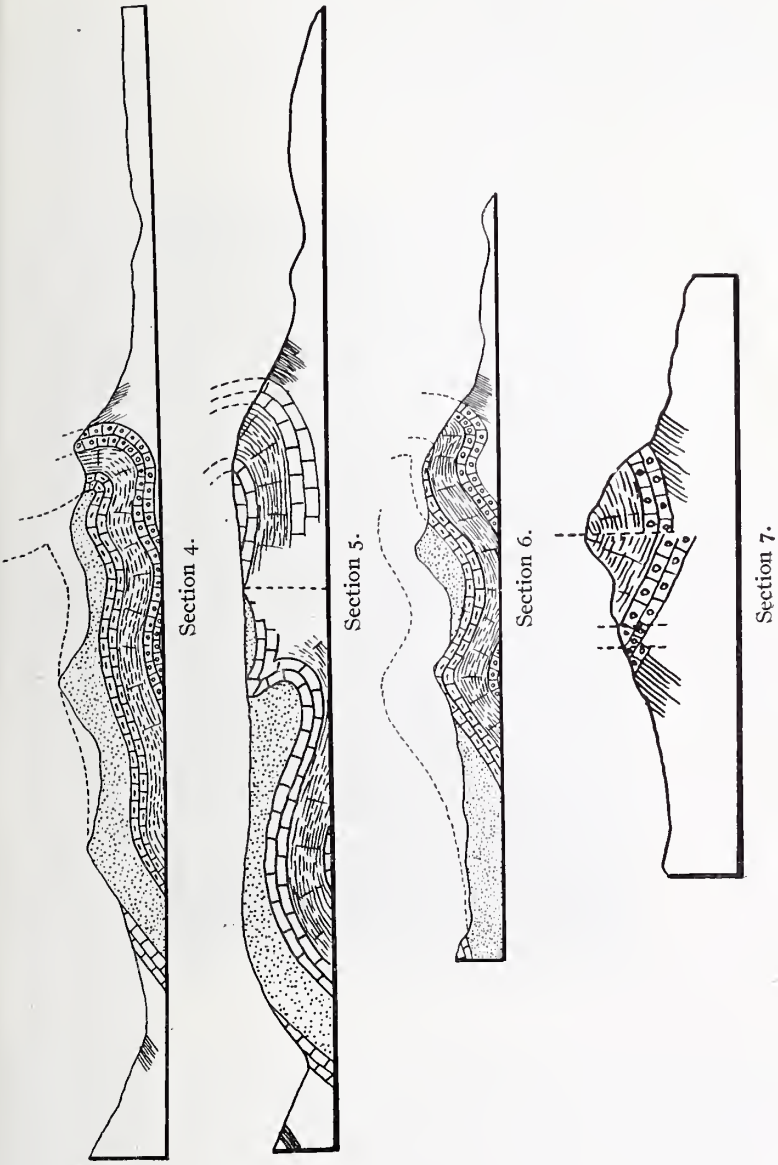
1. Hamilton—Marcellus.
2. Onondaga—Schoharie.
3. Esopus—Oriskany.
4. Port Ewen.
5. Becraft.
6. New Scotland.
7. Coeymans.
8. Manlius.
9. Normanskill.



Section 2.



Section 3.


Section 4.
Section 5.
Section 6.
Section 7.

certain instances thrown worthless material over a productive bed resulting in an excess of overburden (Fig. 4). The folds of the Black Lake region are not as pronounced and towards the north gradually pass into an almost level deposit. This flattening out

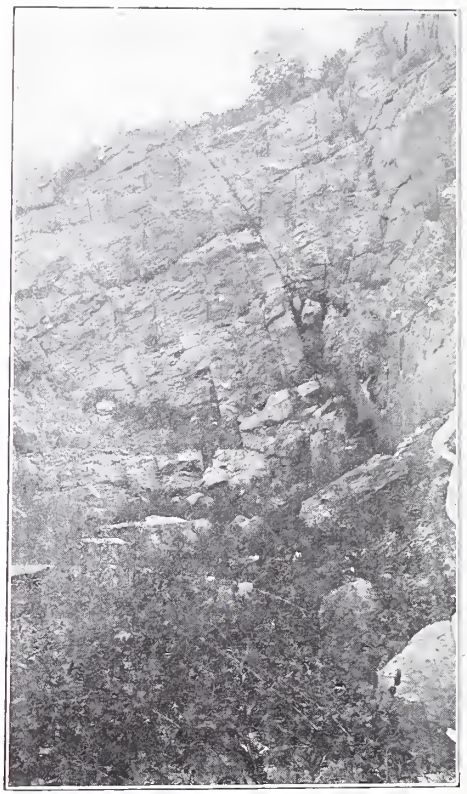


FIG. 2

towards the north is responsible for a better general condition as regards quarry locations. At the north, moreover, the supply of material is greatly increased by the greater area of the Onondaga limestone.

Throughout the southern section of the Van Luven Lake area

the supply of high-grade material is absolutely limited; through the northern section of the same area it is limited by the methods of mining. With the introduction of underground mining, especially in the region between sections 3 and 6, inclusive, there

FIG. 3.

can be an unlimited production. There are many facts brought forward in favor of open-cut mining as compared with underground mining and yet in this region many of the present difficulties as encountered in open-cut mining could be forgotten and a steady production made throughout the year. The possi-

ble high cost of drilling would be cut by the steady production of a high-grade material, with no sorting, no handling of waste

FIG. 4.

rock, no loss of time during the winter months, and no handling of overburden.

TOMPKINSVILLE, N. Y.

THE CLEANING OF SHEET STEEL AND IRON FOR ENAMELING PURPOSES.[1]

By R. R. Danielson.

It is the consensus of opinion among enamelers that one of the prerequisites for successful enameling is cleanliness, especially as applied to the cleaning and pickling of the metal previous to application of the enamels. The term cleaning, as used by platers, however, is a comparatively new one to enamelers, whose recognized name for this same operation is embodied in the processes known as scaling and pickling. The word cleaning can reasonably be used to designate the operations necessary to remove the grease and the oxide of iron from the metals.

While the importance of this subject is thoroughly recognized by enamelers, comparatively little work of a systematic nature has been carried on, and in general the pickling or cleaning department runs according to the judgment of the pickler. Research work of this kind demands commercial conditions for true comparative results, and it is our intention merely to present some data on the various methods of cleaning and pickling as a guide for systematic investigations.

Cleaning may be divided into the following processes:

1. Removal of grease and dirt.
   - (*a*) Scaling.
   - (*b*) Cleaning compounds.
2. Removal of iron oxide.
   - (*a*) Sand blasting.
   - (*b*) Pickling with acids or acid compounds.
   - (*c*) Electrolytic pickling.
3. Neutralizing baths.
4. Drying of ware.

1. Removal of Grease and Dirt.

During the forming operations, especially in the case of sheet metal products, the metal becomes covered with grease and oil.

[1] By permission of the Director, Bureau of Standards.

If allowed to remain, these retard the action of the acid during pickling, and also cause trouble during the subsequent enameling process.

Scaling.—Under the old-established methods the scale is removed by the so-called scaling process, in which the ware is placed in a muffle furnace and heated at a temperature of about 1400° F for a period of from 5 to 10 minutes. To assist in the formation of a light scale which will separate easily from the metal, dilute acid, usually the discarded acid from the pickling tanks, is sprinkled on the ware just before it is placed in the furnace. Where the metal has rusted, this scaling method is especially advantageous, in that it tends to loosen the rust and facilitate the subsequent pickling.

The chief objection to scaling is the tendency of the metal to warp, due to the extra heating beyond that necessary in the enameling process, in the handling of this operation. This is especially true in the case of large, flat sheets. A second but minor objection is the formation of a heavy coating of oxide on the metal which may have been subjected to little mechanical working and hence no annealing, and which, therefore, is covered with only a slight film of oxide common to sheets fresh from the mills. This additional coating of oxide necessarily adds to the time required for pickling. For these reasons, and also as a possible source of saving in manufacturing costs, a large number of manufacturers have experimented with the so-called chemical cleaners with varying degrees of success.

Cleaning Compounds.—Analyses of a large number of chemical cleaners at the Bureau of Standards have shown them to be composed of one or several of the following materials:

Sodium hydroxide.
Sodium carbonate.
Ammonium carbonate.
Borax.
Trisodium phosphate.
Sodium silicate.
Resin in the presence of caustic, forming soap.
Soaps.

Other cleaners used, especially in the japanning industry, are naphtha and gasoline. It is questionable whether these will serve the enameling industry, since they leave a light film of oil over which the water-suspended enamel will not flow evenly.

Another disadvantage of gasoline or naphtha is their inflammability, which makes their use undesirable in any industry.

The alkaline cleaners acting on a fatty oil should combine chemically with them, forming soaps. According to the explanation of soap chemists these soaps act on the remaining oil in a mechanical way to lessen the surface tension, thus separating the greases from the metal.

In the case of mineral oils, which are the basis of the usual lubricating oil, the action is principally a mechanical one. All the chemical cleaners lessen the surface tension and separate the oils from the metal. The globules rise to the surface of the bath where they may be skimmed off and then reclaimed for further use. Therefore, in removing mineral oils, where care is taken in separating the oil from the bath, the latter should serve for a period of weeks and even months with slight additions of cleaner from time to time. Eventually the accumulation of greases and dirt in the bath will limit its usefulness, and this, of course, is dependent on the conditions existing in the individual plant.

The following data were obtained in one enameling plant, using a cleaning bath over a period of approximately three and one-half weeks.

1st week.	New solution, 200 lbs. of cleaner in a 400-gallon tank.
	2nd day, no additions.
	3rd day, 5 lbs. cleaner.
	4th day, no additions.
	5th day, 15 lbs. cleaner.
	6th day, no additions.
	Total, 20 lbs. addition.
2nd week.	7th day, 15 lbs. cleaner.
	8th day, 20 lbs. cleaner.
	9th day, 12 lbs. cleaner.
	10th day, no additions.
	11th day, 12 lbs. cleaner.
	12th day, 2 lbs. cleaner.
	Total, 61 lbs. addition.
3rd week.	13th day, 7 lbs. cleaner.
	14th day, 12 lbs. cleaner.
	15th day, 18 lbs. cleaner.
	16th day, no additions.
	17th day, 15 lbs. cleaner.
	18th day, 2 lbs. cleaner.
	Total, 54 lbs. addition.

4th week. 19th day, 12 lbs. cleaner.
20th day, 20 lbs. cleaner.
21st day, no additions.
Total, 32 lbs. addition.

On the twenty-second working day a new solution was made up and the old solution was used as a preliminary bath for some exceptionally greasy ware. During this period approximately 2500 to 3000 sq. ft. of steel were handled each day. The original solution was made up of about one-half lb. of cleaner per gallon of water with an average addition each week of about one-sixth of this amount of cleaner. These data approximate the usual amounts of cleaner required for cleaning steel for enameling purposes, but exact amounts required can be determined only by experiment under actual conditions.

A prime necessity in the use of these chemical cleaners is that the bath be kept close to the boiling point, which is best secured by placing steam coils in the tank. Agitation, obtained either with a mechanical arrangement or by the introduction of a steam jet, accelerates the cleaning and is really essential to efficient work. It has been found that the use of electric current also accelerates the work, the time required for cleaning being remarkably lessened and better results being obtained. The general explanation for this is that the ware being made a pole, there is a generation of gas bubbles which act mechanically to carry off the grease. It is recommended by writers on this subject that the current be reversed, that is, the material to be cleaned be made the anode. One enameling plant has been very successful with this method of grease removal and has entirely eliminated scaling.

Another possible use of these cleaners is in the removal of enamel from pieces spoiled in the enameling process. The following data were obtained from work carried on in several enameling plants on tests of this kind:

(*a*) Two pounds of cleaner per gallon of water removed a vitreous enamel from kitchen ware in 36 hours without current.

(*b*) Two pounds of cleaner per gallon of water, bath kept at boiling point for 24 hours, removed a vitreous enamel in splendid shape.

(*c*) One-half pound of cleaner per gallon of water, with tempera-

ture of bath 180° F and reverse current at 6 amperes, removed a white enamel in 6 hours.

In these methods of removing enamel there is a possibility of considerable saving in the reclaiming of rejected ware in enameling plants, especially at the present price of steel and the high cost of labor in forming ware.

2. Removal of Iron Oxide.

Sand Blasting.—The next step in the cleaning process is the removal of oxide from the metal. The usual method of removing the oxide from large, sheet iron shapes, especially of heavy gauge, and from cast iron ware, is by means of a sand blast. This is a mechanical method in which the oxide is abraded from the surface of iron, which is left rough as compared with surfaces cleaned with acids. Whether this rough surface is better for enameling purposes is wholly theoretical, as both smooth and rough surfaces appear to enamel successfully in practice.

The use of steel shot and crushed steel is taking the place of sand for so-called sand-blasting purposes. Its chief advantage is the elimination of dust during the operation.

For cleaning metal for enameling, the sand-blast method has an advantage over pickling. The presence of acid in the metal during enameling is harmful, in that it causes blistering during the firing of the enamels. Sand-blasting also eliminates scaling or cleaning as it removes the slight film of oil and dirt as well as the oxide. It is, however, questionable whether sand-blasting on light-gauge metals or irregular shaped pieces would be successful.

Pickling.—Pickling is the process of chemically removing oxide from the metal so that the metal itself is exposed. It is usually accomplished by the use of sulphuric or hydrochloric acid solutions of concentration of 2 to 15 per cent by weight and at a temperature of from 25° to 80° C. Other reagents for pickling include hydrofluoric acid, and solutions of sodium acid sulphate or so-called niter cake.

It is the common theory that pickling is accomplished by the reaction of the acid on the iron or steel with the formation of an iron salt and hydrogen gas. The acid works its way under

the scale by dissolving a thin layer of metal, thus loosening the scale. The final separation of scale is sometimes aided by the pressure of hydrogen-gas bubbles formed during the solution. This action can be plainly seen on any piece of metal covered with a loose scale. Strong acids will dissolve the oxide to a certain extent but weak acids act on the iron alone, allowing the oxide to settle in the tank bottom as a brown sludge.

For most efficient action in pickling, considering minimum attack on sound metal, the acid strength should run between 2 and 4 per cent for sulphuric acid and about 3 to 5 per cent for hydrochloric acid. These limits represent maximum activity on average light scale, but where a heavy scale is encountered it may be necessary to increase the acid content to 10 per cent.

One of the main objections to a strong acid for enamel ware is the tendency for the metal to form blisters, either during the pickling process or during subsequent enameling operations. This is especially true of steels containing impurities. C. Treischel[1] states that a reduction in the acid content from 10 to 3 per cent, using sulphuric acid, decreases blistering enormously, in some cases from 40 to 2 per cent.

That a considerable saving in acid can be realized without materially cutting the time of scaling of ware covered with a normal amount of oxide is shown by the experience at one plant. Moreover, the quality of pickling was found to be much improved with the decreased acid content. The solution bath previous to tests contained an average of from 4 to 8 per cent of acid. To a tank containing approximately 300 gallons of water was added 104 gallons of 1.18 sp. gr. hydrochloric acid, giving an acid strength of 8 per cent. On the second day about 26 gallons of acid were added, bringing the acid content to about 6 per cent. No acid was added on the third day during which the acid content averaged 4 per cent, after which a new solution was made.

It was afterwards found from some trials that 52 gallons of acid to 300 gallons of water and an additional 26 gallons the second day kept the acid content at from 4 to 5 per cent, after which the tank was emptied. This gave a more uniform run of

[1] C. Treischel, *J. Am. Ceram. Soc.*, **2**, 774 (1919).

pickling at a saving of about 25 gallons of acid per week and a decrease in blistering during enameling.

During the last year or two necessity for saving acids and their increasing costs brought to the attention of manufacturers using the pickling process a substitute in the form of niter cake—a by-product of nitric acid manufacture.

Niter cake is an impure acid sulphate of sodium carrying a variable excess of sodium sulphate. Its acidity usually ranges from 28 to 44 per cent sulphuric acid, although the usual content is around 32 per cent. The cake usually contains some impurities such as small amounts of iron, alumina and silica and occasionally traces of arsenic or nitric acid.

Pickling with niter cake does not differ much from acid pickling, except that for best results it is essential that the bath be kept close to boiling temperature. In annealing practice it has been found advantageous to use a mixture of niter cake and sulphuric acid especially where speed in pickling is desired. The pickling capacity of 1 pound of niter cake is equivalent to 0.37 pound of 66° B. sulphuric acid. A mixture of niter cake and sulphuric acid is recommended by Corbett[1] for normal production, although in some cases the acid can be entirely replaced by niter cake. This agrees with the experience of one plant where successful pickling was accomplished by the use of 1 pound of niter cake per gallon of water kept at boiling temperature. The work, however, was principally on metal covered with very little oxide, as it had previously been put through a cleaning solution instead of the usual scaling process.

The following data, the result of successful tests over a period of several months, give some idea of a mixture suitable for enameling-plant pickling.

A typical run was as follows: The bath was made of 700 gallons of water with 275 lbs. of 66° B. sulphuric acid and 100 lbs. niter cake. This gave an acid content of approximately $5^1/_2$ per cent. The bath was then maintained at about this strength by the addition of about 30 lbs. each of sulphuric acid and niter cake daily. It is possible that the actual daily require-

[1] E. E. Corbett, "Pickling with Niter Cake," *Automotive Industries*, Dec. 26, 1918.

ment was high—due to the condensation of steam in the bath. This bath was used for a week when a new charge was made up. During this time approximately 3500 sq. ft. of metal was pickled each day. This bath was very active and ware that required from 10 to 30 minutes in a hydrochloric-acid bath of 6 per cent acidity was put through this mixture in from 2 to 4 minutes with excellent results. One disadvantage of niter, especially when used alone, is its short life as compared with acid. This is due to the addition of sodium sulphate as an unavoidable component of niter cake, which rapidly increases the solids in solution. According to Corbett,[1] it is wasteful to attempt to strengthen baths which have a specific gravity higher than 1.26 to 1.28, as acid at this point carries such a large amount of salts in solution that the activity of the acid has ceased.

The most satisfactory method of testing a pickling bath for acid strength is by titration of a 10 cc. sample of the acid against a normal solution of sodium hydroxide. The formation of a small permanent iron-hydroxide turbidity is taken as the end point. Each cubic centimeter of standard alkali solution represents approximately one per cent of free acid by volume.

The advantages of niter-cake solutions are saving in cost of pickling and absence of acid burning and etching when used. It is a material more easily handled than the sulphuric acid and if kept in a dry place will retain its original content of acid fairly well.

Essential conditions for efficient pickling are agitation and heating of the bath. Reactions are hastened if the temperature of the pickling bath is maintained at 160° to 180° F, and in the case of the niter cake boiling is highly desirable. The average enameling plant makes little use of agitation, although the sheet mills make use of it in all its various forms. The general methods of agitating are by means of steam outlets in the bath or by mechanical agitation of the ware or solution.

Steam agitation is wasteful because of the condensation of water in the bath and the difficulty of getting steam outlets to stand up in use. In mechanical agitation the ware may be raised and lowered in the bath by means of a steam or air hoist or the acid

[1] *Loc. cit.*

level may be raised and lowered by a plunger device in the bath. The former is used where the charge of ware is comparatively light and the latter where heavy charges are carried in the bath. Some modification of these methods could be easily applied to the enameling process, especially in those plants where standard shapes are put through, with a considerable saving in cost of pickling.

In order to eliminate the disagreeable fumes from pickling baths, use has been made of such materials as sugars, flours, soap-bark, etc., the material being sprinkled over the surface of the bath. These organic substances, due to their charring by the acid, form a blanket of charcoal and foam over the acid which retards the expulsion of the hydrogen gas and acid fumes. It is claimed by manufacturers of some of these compounds that the retention of the hydrogen gas in the bath accelerates pickling but experience of picklers apparently shows the contrary to be true. In a number of cases the use of these materials has been discontinued, due to the "killing" of the acids after their use.

Electrolytic Pickling.—This has been used to a considerable extent in the plating industry where currents of low amperage and voltage are obtainable. In this method the work is accomplished in a bath of a suitable electrolyte through which the current is passed. The ware to be pickled is placed as the cathode or anode in the bath. According to Rawdon,[1] when the metal is used as the cathode, the hydrogen liberated reduces some of the oxide and also aids mechanically in flaking it off. When the steel is made the anode, the oxygen liberated mechanically detaches the oxide, which later might dissolve in the electrolyte.

Thompson[2] states that work which required 43 minutes for pickling in a 4 per cent sulphuric acid bath at boiling temperature was pickled in an electrolytic bath in 5 minutes with a current of 9.5 amperes and 2.7 volts.

He states the advantages of electrolytic over chemical pickling as follows:

[1] H. S. Rawdon, Bur. Standards, *Circ.* **80**, "Protective Metallic Coatings for the Rustproofing of Iron and Steel."

[2] M. D. Thompson, *Trans. Am. Electrochem. Soc.*, **31**, "Electrolytic Pickling of Steel" (1917).

1. Speed of pickling.
2. Saving in cost over acid—estimates cost at $^1/_4$ that for acid.
3. Evenness of pickling, as the metal is not attacked, the only action on the metal being mechanical, the chemical action being confined to the scale.

One stove-manufacturing company has experimented with current in their pickling baths. They found that a current of 6 amperes in a bath of sulphuric acid (1 part acid to 4 parts water) cut the time of pickling with a saving in cost of acid. The enameling industry has not yet made use of this method of pickling to any large extent, but from results obtained in other industries, it should be worthy of investigation.

Neutralizing Baths.

In all but gray-ware enamels, it is very essential for successful enameling that all traces of acid be removed from the metal. The mottling in the so-called gray or granite ware is accomplished by the rusting of the metal due to the presence of iron sulphate, so that no attempt is made to neutralize the acid, the ware simply being passed through rinse waters and then dried, or left under water until ready for dipping. The iron sulphate in the presence of air forms iron hydroxide, which appears as rust spots on the metal. In the burning of the gray enamel these spots change to iron oxide which flux the enamel and cause black spots wherever the rust spots occur. These black spots against the gray background give the mottled appearance.

For all other wares the presence of acid on the steel where the usual ground coat is employed leads to many troubles in enameling, such as irregular draining of the enamel and blistering, so that especial care must be taken to neutralize the remaining acid. This is carried out by immersing the ware in a weak alkaline bath made up by dissolving one pound of soda ash in about 25 gallons of water. A better neutralizing agent is one of the soda-ash soda-caustic compounds found on the market. These compounds consist of approximately 2 parts of caustic soda to one part of soda ash and are much more effective than common soda ash. Similar results should be obtained, however, by adding 12 lbs. of caustic soda and 6 lbs. of soda ash to about

400 gallons of water. A decided advantage of such a mixture, aside from its greater neutralizing power, is that such a solution will be effective over a period of several days whereas an ordinary soda bath will become ineffective after one-half to one day of use.

To facilitate rapid drying of the ware and consequently as little rusting as possible, the alkaline bath is kept close to the boiling temperature.

Drying.

Drying is accomplished by passing the ware into driers heated from 150° to 230° F. These are usually heated by waste heat from the enameling furnaces, gas burners or by steam coils.

Waste heat is usually provided by passing air through a flue between an inner and outer lining in the stacks of the furnaces. A blower is placed between the stacks and driers, pulling the air through the flue and passing it on to the drier. The essential points in this system are: (1) that the stack be properly lined to prevent the products of combustion from passing into the waste-heat flue; (2) that proper aprons be provided over the openings to the waste-heat flue to prevent the products of combustion from being drawn in from over the top of the stack. The products of combustion, if allowed to pass over the drying ware, may deposit soot or cause rusting of the metal, either of which would undo the work of cleaning and pickling.

Gas driers are successful, especially where a conveying system is used through the drier and care is taken to obtain complete combustion. The products of combustion from a smoky gas-flame are as harmful as those considered in the use of waste heat.

Steam coils furnish exceptionally clean drying conditions but drying in this way is apt to be somewhat slower and less thorough when ware with closed beads is being dried.

Summary.

A brief summary of the methods used in the cleaning and pickling of metals brings to view a varied set of processes for accomplishing the work. While enamelers have long realized the extreme importance of careful cleaning and pickling, it is readily seen that much can be borrowed from the practice of related industries with

great possibility for decreased costs and increased output. Where the conditions in plants have permitted, enamelers have taken advantage of these opportunities and have instituted the use of new methods, such as removal of grease with chemical cleaners and pickling with acid substitutes, with very satisfactory results.

Bureau of Standards,
Washington, D. C.

PRODUCTION OF SELENIUM RED GLASS.

By F. A. Kirkpatrick and George G. Roberts.[1]

The production of red glass by the use of metallic selenium and selenium compounds as the coloring agents has been practiced in the United States and in Europe for many years. The methods have been kept secret and only a few manufacturers have succeeded in producing selenium glass of satisfactory quality. The various steps in the process, from the compounding of the batch mixture on through to the annealing of the ware, require very careful control.

In addition to the proper degree of color absorption, each of the different kinds of products, such as lenses, globes, bulbs, sheet glass, etc., must possess those physical properties which permit its use for the intended purpose. For example, a lantern globe must be free from such defects as seeds, stones, and cords, and must have sufficiently low viscosity and fusibility to allow it to be blown in molds easily and quickly. The finished globe must be resistant to rupture on sudden heating and cooling and to the agencies of weathering. In order that it may possess these properties, the glass must contain such constituents as lime, zinc oxide and boric oxide. If a red color cannot be produced in such a glass by the use of selenium, some other coloring agent as copper or gold would have to be employed.

As a coloring agent selenium has certain advantages over copper and gold. It is a cheaper coloring agent than gold. With proper manipulation selenium always gives the desired color while copper-red glass frequently comes from the pot spangled and spotted—due to the different degrees of reduction of the copper compounds. Selenium produces a very pure red but other coloring agents produce red colors which allow light to pass in other parts of the spectrum.

In a preliminary study of the methods of production of selenium glass, several melts were made at the Pittsburgh Laboratory of the

[1] By permission of the Director, Bureau of Standards.

Bureau of Standards. It is believed that the results obtained thus far will be of interest to glass manufacturers.

Two kinds of glass were made, a soft-working zinc-alkali glass and a plate glass. The formula and batch weights of the zinc glass were as follows:

$$\left.\begin{matrix} 0.239\ K_2O \\ 0.408\ Na_2O \\ 0.353\ ZnO \end{matrix}\right\} \quad 2.26\ SiO_2.\quad 0.0145\ B_2O_3 \quad \left\{\begin{matrix} 0.0142\ CdS \\ \\ 0.026\ Se \end{matrix}\right.$$

	On basis of 100 sand.	On basis of 100 batch.
Sand	100.0	53.70
Potash	30.3	16.25
Soda ash	30.3	16.25
Zinc oxide	21.2	11.37
Cadmium sulphide	1.51	0.81
Selenium	1.51	0.81
Borax	1.51	0.81

The formula and batch weights of the plate glass were as follows:

$$\left.\begin{matrix} 0.427\ Na_2O \\ 0.549\ CaO \\ 0.024\ MgO \end{matrix}\right\} \quad 2.62\ SiO_2 \quad \left\{\begin{matrix} 0.015\ CdS \\ \\ 0.020\ Se \end{matrix}\right.$$

	On basis of 100 sand.	On basis of 100 batch.
Sand	100.0	62.6
Soda ash	29.8	18.6
Hydrated lime	27.7	17.3
Cadmium sulphide	1.35	0.85
Selenium	1.04	0.65

The compositions of the raw materials used were as follows:

Material.	Principal constituents.	Per cent.
Sand	SiO_2	98.62
Potash	K_2O	54.05
	Na_2O	2.70
Soda ash	Na_2O	57.32
Zinc oxide	ZnO	98.14
Borax	B_2O_3	49.35
	Na_2O	21.54
Cadmium sulphide	CdS	100.00
Metallic selenium	Se	100.00
Hydrated lime	CaO	70.42
	MgO	2.19

The materials were weighed out in 30-pound batches passed through a 12-mesh screen, and mixed by pouring from one mixing can to another several times. The cadmium and selenium were pre-mixed with a small portion of the batch in order to prevent loss of any considerable portion of these coloring agents during the process of mixing.

The glass was melted in a 30-pound covered "monkey pot," shaped like those used in the factories for blown- and pressed-ware glass. The pot was set in a small down-draft furnace fired by gas and compressed air and brought slowly to a temperature of 1400° C.[1] The batch was then introduced into the pot in three fills, within a period of about three hours. After the filling had been completed the glass was allowed to fine for five hours. After standing three hours longer the pot was "taken down," that is, the door was taken out and the glass allowed to cool to a sufficiently low temperature for working. On account of the small size of the pot the glass could be cooled quickly and was ready for working in a half-hour after "taking down." Two or three hours were required for working out the glass.

Shortly before working out, the melt should be skimmed to remove impurities which have collected on the surface. The skimmed glass is saved and used as cullet in the next melt. Metallic selenium melts at 217° and boils at 675°, forming a red-yellow vapor. Hence it escapes readily from the batch during the melting and fining periods and it is necessary that the pot door be sufficiently covered with wet clay to prevent the escape of the coloring agent. If any fumes at all are observed to escape, it is known that some of the selenium is being driven out of the glass. After the selenium becomes incorporated with the glass, probably in colloidal suspension, it is retained permanently, provided the temperature is not raised above 1400°. It should be noted that the time required for the several operations would be much longer in actual practice where larger pots are used.

The glass was gathered on punties and worked by three methods: (1) Pressing in a mold; (2) Blowing in a mold; (3) Blowing and working "off-hand," that is, without the use of a press or mold. The pressed specimens were discs one inch in diameter

[1] All temperatures given in this paper are in degrees Centigrade.

and one-eighth inch thick, and signal lenses three inches in diameter. The blown specimens were salt shakers. The "off-hand" specimens were bulbs and vases. By the use of these three methods, the manipulations necessary for bringing out the color were determined.

A description of the different steps in these processes, the time required for each step, and the results obtained, are tabulated as follows:

METHOD 1.—PRESSING IN A MOLD.

ZINC-GLASS LENSES.

Time in seconds from instant of gathering.	Step in process.
0 to 2	Glass gathered from pot.
2 to 20	Glass marvered (rolled on iron plate).
20 to 40	Lens pressed in mold.
40 to 60	Lens allowed to cool.
60 to 120	Lens flashed in glory hole.[1]
120 to 130	Lens allowed to cool.
130	Lens placed in lehr.

The temperature of the glory hole was 680° and that of the lehr 580°. After all of the glass had been worked out of the pot, the fire was turned off in the lehr and the lenses were allowed to cool 36 hours. They came from the lehr deep-red in color.

METHOD 1.—PRESSING IN A MOLD.

PLATE-GLASS DISCS, PROCEDURE A.

Time in seconds from instant of gathering.	Step in process.
0 to 2	Glass gathered from pot.
2 to 60	Glass cooled in air-blast to about 400°.
60 to 90	Glass flashed in glory hole.
90 to 100	Glass pressed in mold
100 to 110	Disc put into lehr.

[1] In glass working the term "flashed" means heated and softened in a flame or in a special furnace, under either oxidizing or reducing conditions. The term "glory hole" refers to a small furnace having one or more circular openings, 1 to 15 inches or more in diameter, through which the flames are allowed to escape and in which the ware is heated or "flashed" to soften it for working, to give it a "fire polish," or for some other purpose.

The temperature of the glory hole was 1200° and that of the lehr 500°. After 36 hours in the lehr the discs were removed and found to possess a good red color.

METHOD 1.—PRESSING IN A MOLD.

PLATE-GLASS DISCS, PROCEDURE B.

Time in seconds from instant of gathering.	Step in process.
0 to 2	Glass gathered from pot.
2 to 4	Glass carried to press.
4 to 14	Disc held in mold.
14 to 74	Disc allowed to cool in air.
74 to 254	Disc flashed in glory hole.
254 to 264	Disc placed in lehr.

The temperatures of the glory hole and of the lehr were the same as in Procedure A. The discs were yellow in color. They absorbed the blue light of the spectrum and allowed the other colors to pass.

METHOD 1.—PRESSING IN A MOLD.

PLATE-GLASS DISCS, PROCEDURE C.

Time in seconds from instant of gathering.	Step in process.
0 to 2	Glass gathered from pot.
2 to 4	Glass taken to press.
4 to 14	Lens pressed in mold.
14 to 194	Lens flashed in glory hole.
194 to 204	Lens placed in lehr.

The glory hole and the lehr were at the same temperatures as used in Procedure A. The color of the lenses when taken from the lehr was a faint yellow.

A study of the four working schedules given above shows that a red color was obtained by use of the first two only. In these two procedures the glass was cooled immediately, after gathering, from the melting temperature to a lower temperature of from 400° to 700°. It appears to be this cooling process which brings the selenium into the proper state of dispersion for the subsequent development of the red color by reheating, or flashing. The physical processes which take place are no doubt

similar to those which bring out the color in gold-ruby glass, in which it is supposed that on cooling the glass from the melting temperature some of the gold exists in the form of nuclei, which, on reheating, serve as centers of growth and become large enough to give the visible effect of red color.

METHOD 2.—BLOWING IN A MOLD.

Salt Shakers, Procedure A.

Time in seconds from instant of gathering.	Step in process.
0 to 2	Glass gathered.
2 to 30	Glass blown and worked to shape in iron blocks.
30 to 130	Glass bulb allowed to cool until dark (about 400°.)
130 to 200	Glass bulb flashed in glory hole.
200 to 230	Salt shaker blown and held in mold.
230 to 240	Salt shaker placed in lehr.

METHOD 2.—BLOWING IN A MOLD.

Salt Shakers, Procedure B.

Time in seconds from instant of gathering.	Step in process.
0 to 2	Glass gathered.
2 to 10	Glass blown and worked in iron blocks.
10 to 20	Glass bulb taken to glory hole.
20 to 35	Glass bulb heated in glory hole.
35 to 45	Salt shaker blown in mold.
45 to 70	Salt shaker held in opened mold.
70 to 80	Salt shaker placed in lehr.

The temperature of the glory hole was 1200°. The temperature of the lehr was 500° in Procedure A and 600° in Procedure B. It is noted that in Procedure A the glass was allowed to cool after gathering for a period of 130 seconds, while in Procedure B the period of cooling was only 20 seconds. In the first case a good red color was obtained and in the second only a light yellow color.

METHOD 3.—"OFF-HAND WORK."

Decanter-shaped vases, 6 to 12 inches in height, were made by the following method:

Time in seconds from instant of gathering.	Step in process.
0 to 3	Glass gathered on punty.
3 to 8	Glass-bulb blown and swung to length.
8 to 30	Bulb marvered.
30 to 40	Bulb blown to a larger size.
40 to 45	Bulb swung to length.
45 to 50	Bulb taken to work bench.
50 to 60	Neck cut in bulb.
60 to 75	Bulb swung to length.
75 to 90	Bottom of bulb softened in glory hole.
90 to 110	Vase "gotten to shape" by allowing it to stretch and by pressing the bottom against an iron plate.
110 to 125	Bulb of vase blown to a larger size.
125 to 140	Bottom shaped by pressure of tool.
140 to 150	Vase heated in glory hole.
150 to 180	Bottom again "gotten to shape."
180 to 185	Bottom of vase "stuck up" onto another punty.
185 to 225	Top of vase heated in glory hole.
225 to 230	Vase swung and revolved to straighten neck.
230 to 250	Vase rotated on punty and top of neck flared with tools.
250 to 270	Vase allowed to cool to just below red heat.
270 to 275	Vase broken off punty.
275 to 280	Vase placed in lehr.

The temperature of the lehr was 600° and that of the glory hole 1200°. Vases were made of both the zinc glass and the plate glass, and came from the lehr with a beautiful, deep red color. The plate-glass vases tended to develop a sealing-wax red color, due probably to a crystallization of one of the forms of silica. This result was not obtained in the zinc-glass vases which, on prolonged and intense heating, became only deeper in color. The zinc glass, however, when heated for 30 minutes at a temperature of 900°, became crystalline as do most glasses under such conditions.

It is noted from the schedule that in making the vases the glass was allowed to cool for 90 seconds after the time of gathering, before it was reheated. This allowed the small particles of se-

lenium to assume the proper degree of dispersion at the right time. At this time the glass was only lightly tinged with color, but the color became deeper with the successive heatings and coolings that occurred in the process.

On account of the small size of the pot used in melting, only small amounts of the glass could be obtained free of seeds, stones, and cords. These defects could no doubt be overcome by the use of larger pots.

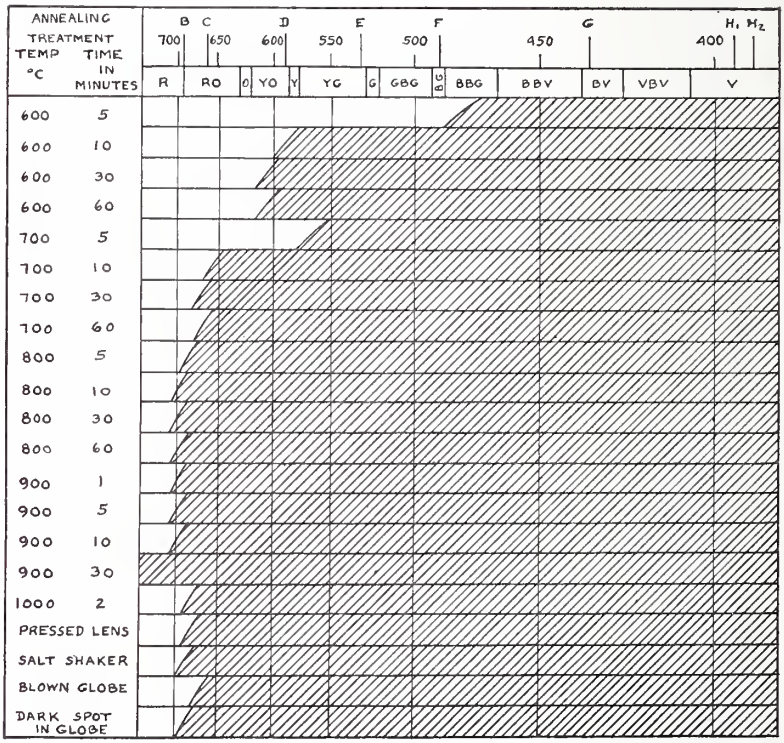


FIG. 1.—Effect of colored zinc-glass specimens on prismatic solar spectrum.

A number of the specimens were examined with a small direct-vision spectroscope. The results are shown in Figs. 1 and 2. The scale at the top represents the wave lengths in millimicrons ($m\mu$), the positions of the Fraunhofer lines, and approximately,

of the colors of the prismatic solar spectrum. The colors and combinations of colors are denoted by the first letters of the words. At the left of the diagrams is given a description of the kind of glass and the treatment received. The shaded portions of the diagrams represent that portion of the spectrum in which all of the light is absorbed, while the unshaded portions represent that part in which the light is transmitted. The line of division between the shaded and the unshaded portions is sloped to indicate a partial transmission of light. Unless otherwise noted the specimens were discs one inch in diameter and one-eighth inch thick. These were made by pressing by the method given under

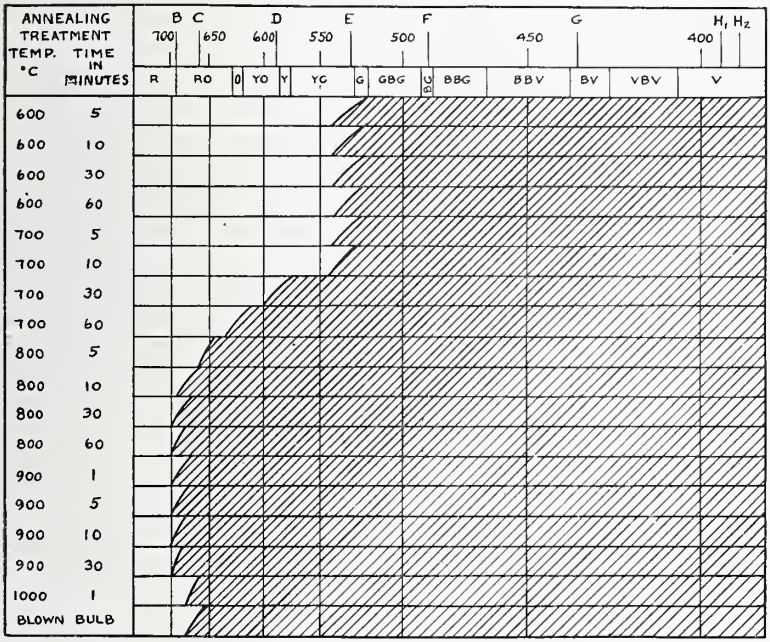


FIG. 2.—Effect of colored plate-glass specimens on prismatic solar spectrum.

"Method 1, Pressing in Mold, Plate Glass Discs, Procedure B." They were then brought slowly to a temperature of 400° and heated in a furnace at the temperatures and for the lengths of time indicated.

In conclusion we may say that the experience gained in carrying out the foregoing experiments has brought to our knowledge several essential features of the process of making selenium red glass. Good red colors were produced in the zinc glass by the use of 0.8 per cent each of borax, cadmium sulphide, and metallic selenium, and in the plate glass by the use of 0.65 per cent of selenium and 0.85 per cent of cadmium sulphide. The two most essential steps in the process are: first, the door of the pot must be securely closed during the filling and fining periods to prevent escape of the selenium by volatilization; second, the glass must be allowed to cool a sufficient length of time immediately after gathering, usually from one-half to one minute.

In the pressed ware the red color was best developed by heating, after pressing, for a period of one to five minutes at a temperature of 900°. The ware blown in molds sometimes developed its color before being placed in the lehr, and at other times after being placed in the lehr. The "off-hand" ware developed its color during the working before being placed in the lehr.

There was brought forcibly to our notice the importance of the method of working, in producing selenium red glass. The composition of such glasses had been known for a considerable length of time but success in making the glass was attained only by complete practical experiments in which were carried out all of the processes of manufacture from compounding the batch mixture to the final annealing of the ware.

Bureau of Standards,
Washington, D. C.

CERAMIC ABSTRACTS.

General.

Acid test for brick as a road material. W. Fawcett. *Brick Clay Record*, **55**, 666–7 (1919).—The U. S. Bureau of Public Roads has started investigations to determine whether a limit should be placed on the size of motor trucks, whether improvements of springs or redistribution of weight is desirable and whether for the salvation of our roads the larger-size pneumatic tires should be substituted for solid tires. For initial tests a novel apparatus which registers the impact of the front and rear wheels of a truck passing over it is used. With the above data in hand it is their object to exert upon a road surface precisely the same kind of impact that is exerted by motor trucks. The machine embodies an unsprung weight together with a sprung weight just as in the case of a motor truck. The entire weight is raised by means of a cam and is allowed to drop a great number of times so that the impact will be exerted on the same spots. Continued impact will be kept up until noticeable failure occurs and it will be possible to deduce accurately just how many vehicles per day any designed type of road will withstand when laid on a known kind of sub-grade. H. G. Schurecht.

How hand-molded brick are made in England. J. W. Overend. *Brick Clay Record*, **55**, 681–3 (1919).—Although machinery has superceded the hand in making brick, there are yet quite a number of firms in England who make brick, chimney-pots, etc., by hand. Clots of clay are taken from the mill by the pug boy to the molders who throw the clay in the mold, which is an oblong box having two sides and two ends, the size being $10'' \times 5'' \times 3''$, in which the brick are roughly molded. The brick are then either sand- or slop-molded. In the sand molding the molds are sanded each time and, since the molder never parts with this mold but sends each green brick away on a pallet, it is termed "pallet-molding." In the other system the mold is dipped into water each time and the mold and brick taken away together.

H. G. Schurecht.

Micaceous or schistose clays. M. S. Sylvester. *Pottery Gaz.*, No. 508, 1078 (1919).—Sylvester, who was commissioned to prepare the highly porous water jars (gargoulettes) at Constantine, Algeria, attempted to prepare same by the addition of sand, sawdust, powdered charcoal and rock salt to the micaceous Constantine earths but was unsuccessful Tests showed that the presence of mica renders the material impervious to moisture when baked and hence porous water jars could not be made from this material but the clay was well adapted for impervious tile. The porous French tile become rapidly

moss grown, owing to their high porosity, whereas the Constantine tile preserve their pink color. This impermeability is more apparent in pottery made from this clay and it is therefore unnecessary to glaze this pottery to make it impervious to water, even with ware of $^1/_8''$ thickness. Argillaceous mica, found in coal, is mixed with excessively plastic clays of poor drying properties and made into brick. H. G. SCHURECHT.

Phenomena of clay plasticity explained. R. F. MACMICHAEL. *Brick Clay Record*, **55**, 677–80 (1919).—The strength of clay, whether plastic, dry or burned, is due to molecular cohesion of the clay particles themselves. The property of cohesion is illustrated by Swedish gauges. These gauges are pieces of hardened steel the ends of which are accurate and true. When the ends of two of these gauges are carefully cleaned and placed together in such a manner as to exclude the air, they will be found to cohere with considerable force (as high as 35 lbs. per sq. in. when tested in a vacuum). If the surfaces are wetted with oil, the gauges fall apart just as water causes clay grains to separate. Plasticity results from the balancing forces of cohesion and disruption. Fluids other than water may be used to produce plasticity in clay. Organic matter, soluble salts and colloids, except as the latter are considered to be merely very fine, insoluble particles of the clay itself, are not essential to plasticity. H. G. SCHURECHT.

Note on forced-draft firing in a periodic kiln. R. K. HURSH. *Clay-Worker*, **72**, 322–4 (1919).—The use of mechanical draft has developed rapidly in boiler and other operations and its use with kilns is now commencing. Mechanical draft may be induced by drawing air through the kiln or by forcing by pressure under the grates. In induced draft the hot products of combustion are handled by the fan, which tends to become deteriorated thereby, although in some cases the gases are cooled before they reach the fan. In forced draft cold air is supplied under pressure beneath the grates and, since the air is cold, the volume of gas to be handled by the fan is less and hence a smaller fan may be used. Mechanical draft overcomes the difficulty of a cold stack and sluggish draft and provides ample draft during water-smoking. The application of forced draft to periodic kilns has, in many cases, reduced both the time of firing and fuel consumption. A heat balance was made on a 26-foot down-draft kiln, using forced draft, air being furnished at a pressure of $1^1/_4$ to $1^3/_4$ inches water gauge. The following results were obtained: (1) The fuel consumption per sq. ft. of grate area per hour was much higher than obtained normally. (2) The greater fuel consumption permitted a more rapid increase of the kiln temperature and allowed the use of a smaller grate. (3) The composition of the flue gases showed large variations in the amount of carbon-dioxide in the gases—probably due to short firing intervals. (4) Carbon monoxide present in the gases was due to insufficient mixing of the air and gases. A larger combustion space above the grate would remedy this to some extent. (5) There was a lag in the temperature of the bottom of the kiln during the early part of the burn but

at the end of the burn the top and bottom temperatures were the same. The following data were obtained:

Number of bricks set	66,000
Weight of bricks set	200.5 tons
Weight of burned bricks	162.5 tons
Weight of coal used	55,835 lbs.
Weight of coal per M bricks	846 lbs.
Weight of coal per ton bricks	343.6 lbs.
Length of burn	123 hrs.
Maximum temperature, top of kiln	1,940° F.

The fuel consumption was below the average for this type of burn—due largely to the short duration of the burn, made possible by the use of forced draft. H. G. SCHURECHT.

Some aspects on ball-mill grinding. L. BROWN. *Brick Clay Record*, **55**, 322 (1919).—Ball mills of different types vary as to efficiency. At the Lenox Pottery cylindrical mills lined with porcelain blocks are used. The mills are six feet long and five feet in diameter and hold a batch of 4,000 lbs. of dry material. The most important factors in grinding are: (1) Effect of time in grinding; (2) Weight of flint pebbles and their wear; (3) Measurement of water; (4) Record of revolutions of mill. Clay substance requires only enough grinding to mix it thoroughly with the coarser ingredients, which must be thoroughly ground before the clay is added. By grinding the clay substance too long it is possible to destroy the working qualities of the body. Indicators to record the number of revolutions are placed on the mills and with the aid of these the time of grinding is kept constant.

H. G. SCHURECHT.

Geological.

Preliminary report on the mineral resources of the United States in 1918. U. S. GEOL. SURVEY. Aug. 7, 1919.—Tabulated statistics of the clay and clay products industry of the United States are contained in this publication which is the first of a new class of Survey reports. E. D. ELSTON.

Refractories.

The magnesite industry in the United States. W. C. PHALEN. *Mining and Sci. Press*, pp. 295–298, Aug. 30, 1919.—Conditions of the magnesite industry are briefly treated. The mineral magnesite is widely distributed in California, occurring in 13 counties, the most important deposits being located in Tulare, Santa Clara, Napa, and San Benito Counties. In the State of Washington there are some deposits in Stevens County. It is interesting to note that the California magnesite is of the amorphous variety and the greater part of it occurs in veins or lenses in massive serpentine.

The thickness of the masses varies from 30 feet to mere stringers and gash veins which are too thin to work. The Washington magnesite is crystalline and occurs as massive beds in sediments consisting of dolomite, shale, and quartzite which have been intruded by basic igneous rocks. The reserves of the Washington magnesite are greater than those of California. The main use of magnesite is as a refractory for lining open-hearth steel furnaces and in lead, copper, and electric furnaces. Some magnesite is used for medicinal and chemical purposes. California magnesite has been employed in making sulphite paper-pulp but dolomite has largely replaced it for this purpose. Other uses are as follows: in fire-resisting magnesia paints and as a non-conducting material for boiler and pipe coverings, also for the manufacture of flooring cement. Crystalline magnesite has long been used for furnace bottoms, the Austrian magnesite containing iron oxide in sufficient amount to make it set well thus forming a hearth capable of resisting to a great degree erosion by molten steel. The Washington magnesite, however, does not contain enough iron oxide, necessitating the addition of this material. Although higher in silica and lime than the Austrian magnesite, the specifications regarding these substances and iron and alumina have been met and the Washington material is now of great importance. The amorphous variety of magnesite is not so satisfactory as the crystalline for refactories, due to the lack of iron. Some of the California magnesite which contained iron oxide in small amounts was used for refractory material but not to any great extent. The main use of the amorphous variety is for flooring materials. Dead-burned dolomite has been used as a substitute for magnesite in refractory furnaces but statistics showing the extent of this substitution are not available. Brief statements concerning the methods of mining magnesite and calcining the ore are given by Phalen and comparison is made between these methods in California and in Washington. E. D. ELSTON.

Qualities, composition and fabrication of silica bricks. *Genie civil,* **74,** No. 2, Jan. 11, 1919.—The best measure of the correct silica content with respect to refractoriness is given by the falling apart of small pyramids, constituted by the material of which the bricks are made plus 6% Fe_2O_3. The life of bricks increases with the SiO_2 content. Al_2O_3 increases fusibility. CaO does not noticeably increase fusibility when used up to 2%. Porosity has great bearing upon the life of bricks, the most desirable results being obtained with a total volume of voids of 18%. Swelling of bricks under influence of heat is greater, the coarser the grain. The rock should be ground so that larger particles pass No. 8 or 10 sieve. Best results are obtained by burning until nearly all the SiO_2 is changed to cristobalite, thus reducing swelling and shifting in the masonry. The writer thinks that the continuous kiln may be suited for burning silica bricks. C. TREISCHEL.

The preparation of raw materials for and the manufacture of glass-house pots. B. J. ALLEN. *J. Soc. Glass Tech.,* **3,** 78–93 (1919).—It is claimed

that inferior fire clays can usually be increased greatly in quality by deflocculation and precipitation of the coarse impurities. Several years ago 21 British fire clays, none of which would rank as grade 1 clay, were thus treated and 17 after such treatment would pass the tests for grade 1 so far as Al_2O_3 content was concerned. An exptl. apparatus for deflocculation and electrolytic collection is described. The ordinary treatment is to blunge 1 kg. of clay with 2 kg. H_2O to which has been added 2–3 cc. of a 10% solution of Na-silicate, NaOH or Na_2CO_3. Blunging takes $1^1/_2$ to 2 hrs., settling 1 to $1^1/_2$ hrs. to remove coarse impurities, settling of the suspended liquor 2 hrs. to allow finer impurities to settle out after which the suspended clay is ready to be collected. By using clays thus treated the use of a special "fusible bind clay" may be avoided. Increased plasticity, gotten by deflocculation and subsequent precipitation, makes use of a larger % grog possible. Grog should be of exactly the same compn. as the raw clay. Grog should be burned hard enough to avoid after-contraction. Black-cored grog should not be used for it causes blow-holes. Grinding should be done in an edge runner mill and a good body of material kept under the runners so the action will be crushing to angular fragments rather than grinding to spherical forms. The ground product should be run over an electromagnet. Sizing is of first importance. Mixing in an edge-runner machine is not satisfactory. Pugging also is inefficient. Slipping is the only way of getting a satisfactory mixture. H_2O with alkali added should be warm when put into the blunger; the clay should be added first, then the fine grog, lastly the coarse grog and blunging should take about 2 hrs. Slip-casting of pots has these advantages, (1) absence of joined surfaces, (2) larger and more grog may be used, (3) uniform H_2O content during drying (4) lower sintering temp. (5) quicker drying. A vacuum applied to the outside of the plaster mold overcomes many of the difficulties of casting and details are discussed. For casting covered pots the vacuum process seems indispensable. Pressure applied on the inside during forming may also be very advantageous. C. H. KERR.

Glass.

Glass making before and during the war. HARRY J. POWELL. *J. Roy. Soc. Arts*, **67**, 485–95 (1919).—Forty-four years ago de la Basties' process of toughening glass by quenching it in oil was discussed at a similar meeting. Glassmaking has been carried on in London for over 300 years. In 1696 there were 24 glass factories in the London district. London's preëminent position was later lost however. Before the European War the English glass industry was alive but not much more. Chance kept alive with difficulty the making of optical glass and some automatic bottle machines had been introduced in Yorkshire plants. There was very little scientific knowledge available and very little provision for its development. The war forced developments in both science and practice. At the Whitefriars plant was melted the special spectacle glasses for Sir William Crookes, other special

glasses, and thermometer tubing equal to the best German product. The author hoped to make thermometer tubing superior to the Jena 16 III and 59 III but the publication of formulas for the pre-war German glasses, which thermometer makers accepted as official standards, has interfered. The effect of free trade on the glass industry was disastrous and figures are given to prove it. After war was declared the making of vases and fancy table ware was stopped and the men and equipment were used for essential wares. A number of Na-Ca-Al glasses were introduced for mine-horns, for X-ray bulbs and for thermometers, also resistance glass in 4 colors for ampoules, enamels for thermometers, sealing flux for X-ray bulbs, blue enamel for cat's eye levels, white opal glass for arc-lamp shades, sheet glass containing oxide of didymium (for Sir William Crookes), sheet glass containing U (for the Admiralty), a neutral glass for acetylene welders, a dense black glass for airmen's goggles, anti-heat and anti-glare spectacle glass containing Ce, glasses and enamels for artificial eyes, dense lead glass for X-ray shields, etc. Efforts of the various concerns were concentrated on the following: Bagley & Co. Ltd., installing Owen's automatic bottle machines; Breffit & Co., jam and fruit bottles and food containers; Burtles, Tate & Co., Ltd., chemical and lighting ware; Edinborough & Leith Flint Glass Co., electric lighting bulbs for miners' lamps and trench and naval signalling, wireless telegraph valve glass, soft soda glass for lamp working and various colored glasses; Edison and Swan Glass Works, electric lamp bulbs, bulbs for half-watt lamps, for wireless telegraph valves, special colored glasses for signalling, a dark blue glass for ship lighting when running with lights out, and opal tubing; Frank Tomey & Co., soda glass tubing for laboratory ware, thermometer glass, etc.; Greener & Co., installing labor saving machinery for making tumblers, etc.; Molineaux Webb & Co., lighting, chemical and medical glass; Pilkington Bros., gas mask eye-pieces, photo plates, glass screens, searchlight projectors, tables for picric acid making, non-actinic glass for aerodrome roofs, blue glass for stern lights etc.; Stevens and Williams, table ware, chemical and medical glass, miners' lamps, electric bulbs and lighting ware; Stewart & Sons, electric light bulbs; Thos. Webb & Sons, lamp bulbs, glass tubing and rod, chemical ware and table glass; North British Glass Works, glass parts for aeroplanes, combustion glass, laboratory glassware, miners' safety lamps, etc., Duro Glass Works, a Na-K-Ca-Al glass for lamp-working and a Zn boro-silicate resistance glass for beakers and other laboratory ware; Wood Bros. Glass Co., electric light bulbs, laboratory and scientific glassware, flash-lamp lenses and X-ray bulbs, food containers; Derby Crown Glass Works, optical glass; Osram-Robertson Lamp Works, electric lamp bulbs; Chance Bros. & Co., optical glass, special colored glasses for sextants, plate glass for transmitting ultra-violet, a specially transparent blue filter, a uranium glass of great intensity of fluorescence, high pressure lamp globes, heat-resisting glass, glass for gas masks and Crooks' spectacle glass. In conclusion the author shows that very complete adoption of machine processes is essential to the future of the English glass industry. C. H. KERR.

The examination of optical glass in relation to weathering properties. Alfred Vincent Elsden, Oswald Roberts and Harold Spencer Jones. *J. Soc. Glass Tech.*, **3**, 52–70 (1919).—On glass parts of optical munitions of war considerable trouble has been encountered in England due to a film or deposit on the glass surface. There seemed to be 2 kinds of films: (1) small, discreet globules easily visible under low power magnification and (2) extremely minute particles, forming a more or less uniform "bloom" but sometimes recognizable under high magnification as having a globular structure. The trouble was especially marked in hot climates. Some of these troubles were due to poor cleaning or to foreign substances getting onto the lenses but some was from the glass itself. Weathering properties depend primarily upon chem. compn. but the nature of the surface is also an important factor. Weathering resistance has been tested quite commonly by heating in an autoclave at temps. up to 180° C with water. The conditions, however, differ so widely from those of practice that results may be questionable for some types of glass at least. Surface solubility tests are not easily made on optical glass because of the form of the product. Powdered samples might be tested but the exposed surface area is uncertain and in optical glass it is very desirable to test the actual polished surface. Zschimmer's method (cf. *Z. Elektrochem.*, **11**, 629 (1905)) of comparing hygroscopicity by heating polished specimens at 80°C in air practically saturated with water vapor and comparing the resulting deposits seemed very promising. The iodoeosin test also deserved further study, the freshly broken surface being compared with a broken surface after seven days' weathering in moist air at 18°. The iodoeosin test gave concordant and valuable results, except when applied to the heavy and complex optical glasses. For the "dimming" tests a special thermostat and auxiliary apparatus was constructed to permit careful control in passing a stream of moist air over the specimens at 80°C. The apparatus is described in detail. Test specimens are preferably polished plates $1^1/_2'' \times {}^7/_8'' \times {}^1/_8''$, and "blanks" of quartz glass are run in every test alongside the glass sample or samples. No quantative measurement of the "dimming" effect seemed possible but classification was attempted with fair success by assigning a value of 1 to samples unaffected or only very lightly affected; 2 affected; 2 decidedly affected; 3 very seriously affected. The table shows the compn. of some optical glasses tested.

Mark.	Glass.	SiO_2.	B_2O_3.	CaO.	BaO.	PbO.	ZnO.	Sb_2O_3.	Al_2O_3.	MgO.	Na_2O.	K_2O.	SO_3.	H_2.	Reputation.
A	Ca-Na-K silicate	73.03	.....	6.91	.....	.....	....	....	0.74	0.53	12.24	4.38	1.35	....	Very poor
B	Ca-Na silicate	74.63	.....	6.95	.....	0.07	0.39	0.37	3.19	0.11	14.51	.....	....	....	Very poor
C	Ca-Na silicate	73.83	.....	7.08	.....	.....	....	....	1.73	0.15	16.59	0.21	....	....	Very poor
D	Ca-Na-K silicate	73.82	.....	7.16	.....	0.12	....	0.30	1.43	0.27	13.21	3.77	....	....	Very poor
E	Ca-Na-K silicate	71.31	.....	10.08	.....	.....	....	....	1.39	0.23	6.70	10.31	....	0.17	Fair
F	Ca-Na silicate	71.70	.....	11.41	.....	.....	....	0.33	1.92	0.29	13.64	0.99	....	....	Fair
G	Ba-Na silicate	67.02	.....	0.86	9.88	.....	2.01	....	0.70	0.35	17.35	1.40	....	....	Very poor
H	Pb-Na-K silicate	65.24	.....	0.57	.....	12.00	2.16	....	1.45	0.36	13.53	4.53	....	....	Very poor
I	Ba-Na-borosilicate	68.22	10.11	0.60	3.19	.....	....	....	1.33	0.32	13.34	2.89	....	....	Good
J	Pb-K silicate	43.09	.....	.....	.....	43.52	....	0.10	0.80	....	2.22	9.89	....	0.22	Doubtful
K	Ba-Pb-Zn-K silicate	56.65	.....	.....	14.75	10.28	4.01	....	0.38	....	2.04	11.93	....	....	Good

The following table shows the dimming test and iodoeosin results with the above and other glasses:

Glass.	Dimming test (30 hrs.) classification.	Freshly broken surface.	Broken surface weathered 7 days at 180° C.
A	3	0.30	2.21
B	3	0.39	0.60
C	3	0.39	0.37
D	2+	0.16	0.25
E	1+	0.13	0.11
F	1+	0.13	0.14
G	3	0.21	0.31
H	2+	0.66	0.97
I	1	0.02	0.04
J	2	0.35	0.36
K	1+	0.13	0.13
Plate	3	0.13	0.27
Hard crown	2	0.25	0.26
Spectacle crown	2+	0.29	0.26
Soft crown	3	0.14	0.51
Medium Ba crown	1	0.14	0.06
Ba silicate crown	2+	0.12	0.35
Ba silicate crown	2—	0.24	0.24
Ba silicate crown	2	0.09	0.06
Ba light flint	2+	0.17	0.59
Ba light flint	1+	0.11	0.12
Ba light flint	2	0.14	0.27
Light flint	1+	0.41	0.16
Light flint	2+	0.11	0.12
Heavy flint	2	0.35	0.36
Heavy flint	1	0.11	0.12
Heavy flint	2	0.43	0.11
Extra dense flint	1+	0.15	0.11

These tests are sufficient to show the great value of such a test as the dimming test. Further work will be done. Some expts. have indicated the possibility of carrying out the test satisfactorily at a lower temp. than 80°C or in a shorter time than 30 hrs. or both. C. H. KERR.

The annealing temperatures of the lime-soda glasses. S. ENGLISH AND W. E. S. TURNER. *J. Soc. Glass Tech.*, **3**, 125–8 (1919); cf. *Ibid.*, **2**, 90 (1918); *C. A.*, **12**, 2240.—Only the most general relationships between annealing temp. and compn. are as yet known. Practical experience has shown that lead glasses are readily annealed while chem. glassware requires a high anneal-

ing temp. and most careful cooling. The glasses now studied cover a wide range of soda-lime glasses including the more important com. glasses. The compns. follow:

Glass.	SiO_2.	Na_2O.	CaO.	MgO.	Al_2O_3.	Fe_2O_3.	As_2O_3.	Total.
1.....	74.05	25.34	0.21	Trace	0.24	0.14	..	99.98
2.....	73.92	23.80	1.50	Trace	0.20	0.14	..	99.56
3.....	74.08	23.00	2.61	Trace	0.21	0.15	..	100.05
4.....	74.07	21.50	3.81	Trace	0.28	0.14	..	99.80
5.....	73.78	20.78	4.50	0.15	0.38	0.19	..	99.78
6.....	73.18	19.38	6.26	0.21	0.58	0.61	..	100.22
7.....	74.41	17.20	7.45	0.24	0.30	0.40	..	100.00
8.....	74.99	16.00	8.16	0.26	0.31	0.09	..	99.81
9.....	74.96	14.88	9.36	0.28	0.42	0.16	..	100.08
10.....	74.59	14.22	10.38	0.30	0.45	0.21	..	100.15
11.....	74.93	13.02	11.68	0.31	0.38	0.17	..	100.49
16.....	66.71	12.72	18.17	0.85	1.41	0.31	0.30	100.47
17.....	69.73	11.22	17.38	0.55	0.50	0.25	0.47	100.10

The complete detn. of the annealing conditions for any glass involves detn. of (1) the upper annealing temp. at which annealing occurs most readily without deformation, (2) the lower annealing temp. below which the annealed glass can be cooled rapidly without strain and (3) the rate of cooling between (1) and (2). Only (1) is considered here. Strain was noted by optical examn. of short rods, 3 cm. long and 8 mm. diameter. The results are averages of 2 detns.

Glass No.	CaO.	Annealing temp. °C.	Glass No.	CaO.	Annealing temp. °C.
1.....	0.21	483	8	8.16	552
2.....	1.50	490	9	9.36	562
3.....	2.61	492	10	10.38	581
4.....	3.81	503	11	11.68	601
5.....	4.50	512	16	18.17	608
6.....	6.26	526	17	17.38	613

Calculating the compn. to empirical formulas and keeping $Na_2O = 1.0$, the annealing temp. varies with CaO equivalent as follows: 0.0 CaO, 480°C; 0.5 CaO, 510°C; 1.0 CaO, 560°C; 1.2 CaO, 590°C. Results show that glasses with high alkali can be annealed at a fairly low temp. and although requiring a higher temp. than lead glasses, they nevertheless resemble the latter in softening readily. With increase in lime the glass hardens and a higher

annealing temp. is required. The ordinary, white flint (lime-soda) glass used for medical bottles, paste, sauce, pomade bottles etc., has a compn. like Nos. 7 and 9. Other constituents if present in appreciable amts. will, of course, modify the required annealing treatment. C. H. KERR.

The exploitation of glass sand in the United States of America. P. G. H. BOSWELL. *J. Soc. Glass Tech.*, **3**, 72–8 (1919); cf. *C. A.*, **12**, 214.—At Ottawa, Ill., the St. Peter's Sandstone of Carboniferous age is worked. The best is used for glass plants. There is only 1–2 ft. of soil overburden. At the largest workings, those of the Ottawa Silica Co., the blasting shatters the soft rock and subsequent immersion in water causes complete disintegration so it is sluiced down and then handled by belt elevator with cross battens. The sand used for glass making has the following typical size of grain: over 1 m/m, 0.5%; 1.0–0.5 m/m, 21.6; 0.5–0.25 m/m, 75.6%; 0.25–0.10 m/m, 1.3%; 0.10 to 0.01 m/m, 0.3%; below 0.01 m/m, 0.7%. At Rockwood, Mich. a very pure sand has lately been worked and used largely for optical glass. The deposit is in the Sylvania Sandstone of Silurian age. The rock is soft and crushes easily. A typical sieve test, in the same size divisions as indicated above, shows the following percentages respectively: 0.0, 2.2, 79.5, 79.5, 18.0, 0.2, 0.1%.

	Ottawa (washed).	Ottawa (unwashed).	Rockwood (not washed).
SiO_2	99.48	98.47	98.00
Al_2O_3	0.16	0.75	0.13
Fe_2O_3	0.02	0.08	0.012
TiO_2	n. d.	0.05	Trace
CaO	0.11	0.21	0.70
MgO	0.05	0.08	0.41
Na_2O	None	None	None
K_2O	None	0.06	None
Ign. loss	0.13	0.47	0.89

C. H. KERR.

Some experiments with a gas-fired pot furnace. MORRIS W. TRAVERS. *J. Soc. Glass Tech.*, **3**, 70–2 (1919).—In order to make about 5 tons of resistance glass per week and to start producing as soon as possible, a furnace based on the well-known type of horizontal gas-fired retort setting was made, using covered pots 36″ x 38″. Producer gas entered the arched space under the siege in each arch and ascended into the arch through two 6″ x 9″ ports in front of the pot. Secondary air, not preheated, met the gas about 8″ below the top of the siege. Thus the gas was burned as near the front of the pot as possible and cracking of pots down the front was cured. C. H. KERR.

Technical education in the glass industry. By a MEMBER OF THE STAFF. *Pottery Gaz.*, No. 507, 956–958 (1919).—A few years ago a Department of Glass Technology was founded at the Sheffield University in which students are being received and a degree may be obtained in glass technology. A special building taking the form of a small works was constructed where it is possible to produce glass on a semi-commercial scale. In the basement there is a special room for storing glass pots at a constant temperature. Open and closed pots are used, the former being made by the Department, while the latter, ranging in size from 28 pounds to 1 1/2 cwt., are bought. Another room contains the meters which register the consumption of gas and electricity, and a number of powerful air compressors. Another room contains grinding and polishing machines and also a special machine for testing the resistance of bottles to internal strains. There is also a batch mixing room containing special bins for storing raw materials. An automatic screen which separates the dry material into three degrees of fineness (greater than 30-mesh, between 30- and 100-mesh, and through 100-mesh), is used. The furnaces are on the second floor, including a pot arch of the Richmond recuperative type, a 56-lb. whirling furnace and a number of small experimental furnaces varying in capacity from 6 to 14 lbs. On the third floor instructions are given in glass blowing where at the present time a large number of wounded soldiers and girls are receiving instructions. H. G. SCHURECHT.

Silica rock and sand for glass making. F. GELSTHARP. *Rock Products*, No. 22, 29 (Oct. 1919).—Sand for use in glass making is of three physical types. (1) Wind or water deposits composed of loose quartz grains. (2) Sandstone rock composed of a soft or hard cemented mass of coarse grains. (3) Quartz or silica rock. The chemical purity of the sand depends entirely on the kind or grade of glass required. The following are chemical and mechanical analyses of sands satisfactory for the ordinary pale green and better grades of glass:

	1.	2.	3.	4.	5.
SiO_2	99.695	99.580	99.500	99.300	98.53
Fe_2O_3	0.025	0.040	0.045	0.109	0.18
Al_2O_3	0.080	0.150	0.225	0.367	0.83
CaO	0.030	0.060	0.030	0.022	0.02
MgO	0.020	0.020	0.020	0.022	0.05
Ignition loss	0.150	0.150	0.180	0.180	0.39
16- to 30-mesh particles (1.13 to 0.50 mm.)	20.3	5.0	3.2	1.4	0.9
30- to 60-mesh particles (0.5 to 0.22 mm.)	62.6	38.2	78.0	52.3	64.6
60- to 120-mesh particles (0.22 to 0.11 mm.)	14.6	53.0	16.3	45.4	30.0
Particles through 120-mesh	2.3	3.8	0.5	0.9	4.5

Fe_2O_3 is the most injurious impurity on account of its strong, color producing ability. Metallic iron, which is equally as harmful, may be introduced by the grinding of silica or quartz rock and hard sandstone-rock. When high-grade glass is required the sand should be passed over a magnetic separator. Al_2O_3 should not be present in large amounts. It is generally present as clay which is high in Fe_2O_3. If properly crushed sandstone is well washed the Al_2O_3 present as clay will be removed. If analysis then shows much Al_2O_3 remaining, it is present in soluble or insoluble silicates, feldspars, or other compounds which may or may not be injurious. CaO and MgO are present in most glass sands in unobjectionable amounts. Some sands contain vegetable matter which must be washed out to make them fit for use in glass. Sand grains larger than 16-mesh or smaller than 120-mesh size are undesirable. The large grains are slow in melting and may leave strings or striae in the glass. Sandstone grains of this size may consist of small grains held together by a cement containing an objectional impurity. The fine grains which pass a 120-mesh screen are not objectionable if they are as pure as the aggregate, but these fine particles usually contain more impurities than the grains of between 16- and 120-mesh size. The most desirable glass sands are those which have the largest amount of grains of size between the 40- and the 80-mesh screens. Sand, all of which passes the 100-mesh screen, is not desirable. It is liable to be carried out with the gases evolved during melting. If mixing is not thorough or the melting not properly carried out, "white knots," composed of balls of fritted silica, are produced in the glass. Also such sand is too bulky, requiring too long a time of filling. The time of melting is decreased by use of finely ground limestone but increased by use of finely ground sand. Fine sand lowers the heat conductivity of the glass batch much more than does the fine limestone since there is much more sand than limestone in the batch. The fluxes attack find sand more readily than coarse sand but the beginning of the action is delayed by the lowering of the heat conductivity of the raw batch. But in the case of the finely ground limestone its effect in decreasing the heat conductivity is greatly overbalanced by the more active fluxing action, due to the greater surface of lime exposed to the sand. The following figures represent the maximum amounts of Fe_2O_3 allowable in sand to be used in making glass for the ware indicated:

Optical and spectacle glass	0.025%
Better grade of flint and soda-lime glasses for pressed and blown ware	0.050%
Plate glass and pale bottles	0.100%
Common dark bottles	0.500%

F. A. KIRKPATRICK.

Enamels.

The enameling of lava. J. DARCHIE. *Ceramique*, No. 375, 145–6 (1919).—The lava of Volvic has been utilized, after being enameled, for many purposes including laboratory tables, hospital tables, baths, monuments, decorative

purposes, etc. The lava is mined in blocks, sawed and trued to the required shape and the porous surface is filled with a special composition. The enamel is applied by dipping, spraying or with a brush and the ware is then fired in muffle kilns. H. G. SCHURECHT.

Cement and Lime.

Discussion—The causes of unsoundness in portland cement. G. A. RANKIN. *Concrete, Cement Mill Section*, **15**, 33 (1919).—Discussion of article by B. F. Erdahl (*J. Am. Ceram. Soc.*, **2**, 673) restates Mr. Erdahl's summary as follows: (1) CaO is not the cause of unsoundness. (2) $2CaO.SiO_2$ is the cause. (3) Underburning produces a so-called "dusting" clinker and unsound cement. (4) This dust is more or less a disintegration product of an unstable $2CaO.SiO_2$. These points are discussed by Rankin as follows: (1) Microscopical examination of finely ground cement has shown that a grain of cement, even so small that it will pass a 200-mesh sieve, may consist of a perfectly sound shell, free from cracks, and enclose one or more particles of free lime. Such particles may be evenly distributed throughout the cement, and will ultimately cause unsoundness. The hydration or absorption of water by the shell of the cement grain may require a long time, and if, as generally happens, the water does not penetrate to the lime before the set, the cement will be unsound. Such cement is either under-burned or over-burned. Unsound cements of this nature may, of course, at times be made sound by sufficiently fine grinding. Mr. Erdahl presents data to show that as much as 15% CaO may be mixed with a sound cement without causing unsoundness. But in these experiments, the CaO gradually hydrated during the gauging of the cement, was completely hydrated before the cement set, and hence did not cause unsoundness. (2) $2CaO.SiO_2$ does not change chemical composition on dusting. This phenomenon is due to a 10% volume expansion which accompanies a change from a high temperature crystalline form to a low temperature form. All properly burned portland cement contains 25 to 35% $2CaO.SiO_2$ in the particular form which dusts under certain conditions. Fe_2O_3, alkalies, etc., prevent dusting if the clinker is burned at a sufficiently high temperature. Such clinker does not dust or disintegrate when ground and mixed with water. If this were not so, no cement structure could be considered safe. $2CaO.SiO_2$ in the "dusted" form is a very inert material, which does not hydrate readily, nor undergo any changes which could produce unsoundness. It is as so much sand, provided the $2CaO.SiO_2$ has completely "dusted." All commercial cements contain at least a small amount of "dusted" $2CaO.SiO_2$ and evidently "dusting" is generally complete by the time the cement is ground and made into pats. If this were not true, all cements would be unsound. The discussion is continued by Mr. P. H. Bates as follows: The question of soundness is generally misunderstood. When water is added to continual cement the products of hydration produce both an expansive and a cohesive force. The former may predominate with resultant disintegration. In the great majority of cases, however, the latter

exists in the greater amount and the product has cementitious properties. Which will predominate at any time, after gauging, depends upon the amount of certain constituents at the time of gauging. If a cement contains $^1/_2\%$ of the constituent producing expansive force and a low % of slow hardening (not slow setting—setting and hardening must be carefully distinguished) constituent, then at the end of 24 hrs. this cement may be unsound according to the boiling test, while a cement having the same amount of the constituent producing the expansive force, but a larger percentage of quick hardening constituent, would be sound. Furthermore, the first cited cement at the end of 48 hrs. after gauging, may be sound according to the boiling test. The changing of a cement from unsound to sound by storage in bulk is due to an increase in the amount of the expansive constituent and a consequent increase in the % of the cohesive constituent. If immediately after such a change has taken place some of the cohesive constituents are modified and changed to quick setting (and in this case slow hardening) constituents, by the use of a few tenths of a per cent of Na_2CO_3, the cement will again be unsound. This is due to the fact that the relative amount of the constituent producing the expansive force in hydrating has been changed. The constituent which produces the expansive force is free CaO. The amount of this necessary to produce unsoundness will depend upon the amount of quick hardening constituents, which are $3CaO.SiO_2$ and to a lesser degree $2CaO.SiO_2$ (stable form). That the "dusting" clinker referred to by Erdahl was unsound was due to the lack of quick-hardening constituents and not to the "dust," except insofar as it reduced the amount of the stable $2CaO.SiO_2$, that form of the silicate which is alone capable of hardening. For the same reason, the non-dusting material was sound, notwithstanding the fact that it may have contained the same amount of free CaO or even more. The discussion is continued briefly by other writers who agree with Mr. Rankin and Mr. Bates. Mr. Erdahl replies that in a future article he will give further evidence to prove his points, that he will discredit the embedding theory and show that it is the $2CaO.SiO_2$ which does not dust before the pat is set that is dangerous. F. A. KIRKPATRICK.

Formation and properties of blast-furnace slag and portland cement. *Genie civil*, **73**, No. 26, 512–3 (1919).—*Blast-furnace slags:* All blast-furnace slags contain from 5 to 15% Al_2O_3. Within the range of the said limits one can observe the following facts as regards lime and silica: The differences in the conditions of operation of blast-furnaces fired with wood and those fired with coke manifest themselves by corresponding differences in the composition, fusibility, and fluidity of the slags found in the two cases. In the wood-worked furnaces, for an Al_2O_3 content of the slag between 12 and 15%, the SiO_2 percentage varying between 40 and 55%, one finds that the minimum temperature at which the slag has a water-like fluidity lies in the neighborhood of 1350°C, in the presence of 45% SiO_2, and further that the melting point changes very little between the before-indicated limits or values. In the coke-fired blast furnace, the elimination of sulphur in the ashes makes it

necessary to increase the proportion of the bases, especially of CaO, which is distributed between the SiO_2 and the S in an up to the present none too well-known manner. The S would seem to become eliminated so much better the richer the slag in CaO; on the other hand, however, the higher the CaO content of the slag, the lower its fluidity. Slags with high-CaO contents flow while forming isolated drops, similarly as fused lead, while slags rich in SiO_2 flow in elongated drops or sometimes thread fashion like glass. The temperature at which markedly calcareous slags (with 13.50 to 23% CaO) begin to show ready fluidity is not much higher (20° to 25°C in fact) than the temperature at which they begin to grow soft, whereas it is from 200° to 300° higher than the softening point of very silicious slags. An examination of the fluidity diagrams shows that in the presence of great quantities of SiO_2 in the slag, say up to 60% and over, as results from wood firing, the said value may vary within very wide limits, by 15% approximately, without either the fluidity of the slag or the working temperature varying in a marked degree. Wholly different are the conditions relative to coke firing. In the presence of a content of 34% SiO_2 ($CaO:SiO_2=1.5$), the working temperature must be 200° higher than the temperature of softening, if the fluidity shall be adequate. Raising the SiO_2 content to 37% ($CaO:SiO_2=1.35$) the fluidity is much more marked, but desulphurization is incomplete. If the SiO_2 percentage be reduced to 32, the working temperature of the blast furnace must be raised notably, and this means a greater consumption of fuel so much more as also the quantity of slag becomes greater. If the necessary quantity of heat were not furnished, the result would be that the slag becomes pasty and the desirable desulphurizing effect, caused by high CaO contents, would largely be lost. These conclusions agree with those found in actual practice, although blast-furnace slags contain MgO, oxides of manganese and iron, as well as CaS_2, besides the said system $CaO–Al_2O_3–SiO_2$.

Portland cement: Diacalcium silicate, $2CaO.SiO_2$, exists in three different forms or phases, *viz.*, the α phase melting around 2130°; it passes at 1420° into the β modification, and at 675° into the γ modification, with the transformation being reversible. The change from the beta into the gamma modification is associated with an increase in volume. The presence of the beta phase in portland cement is one of the causes underlying its destruction by pulverization. The properties of clinker of portland cement could be rather easily ascertained were it solely constituted by combinations made up of CaO, Al_2O_3, and SiO_2, and if the whole had really passed through a state of complete fusion whereby homogeneity of the mass would be insured. However, the trouble is that often merely sintering, in certain portions at any rate, and incomplete fusion occur; and, apart from the three components $3CaO.SiO_2$, $3CaO.Al_2O_3$, and $2CaO.SiO_2$, regarding the existance of which scientists are practically unanimous, one finds also in the clinker free or uncombined CaO and the compound $5CaO.3Al_2O_3$ which result from an incomplete combination of the constituents. At all events, we may say that good cements of the usual grade as maufactured industrially should contain at least 90%

of the three components above referred to, while the balance of 10% is ordinarily made up of MgO, iron oxides, alkalies, etc. According to Rankin the formation of clinker would occur as follows: First, carbonic acid gases are given off as originate from the calcination of carbonate of lime employed as the raw material to furnish the CaO. After that, the two compounds, $5CaO.3Al_2O_3$ and $2CaO.SiO_2$, are very rapidly formed one after the other, which at once fixing additional quantities of CaO, result in $3CaO.Al_2O_3$ and $3CaO.SiO_2$. These latter two compounds, contradistinct from the two before-mentioned, form very slowly. Their formation is accelerated if brought about inside a molten medium, a flux, which promotes contact-making between solid molecules. Such a flux is formed rather rapidly, namely, the eutectic $2CaO.SiO_2$-$5CaO.5Al_2O_3$-$3CaO.Al_2O_3$ which melts at 1335°C. If the temperature goes beyond this point the formation of the compound $3CaO.Al_2O_3$ is hastened, and then $3CaO.SiO_2$ forms in turn from CaO and $2CaO.SiO_2$. In practice, the reactions are there stopped because one invariably keeps below a temperature at which the entire mass would be in molten state. In brief, in a measure as the temperature is raised, one is dealing with, or there are present, the following combinations (Table 1):

TABLE 1.

	1000°.	1000°–1335°.	1335°–1450°.	1450°–1650°.	Clinker solidifier.
$CaCO_3$	CaO	CaO	CaO	CaO	$2CaO.SiO_2$
Al_2O_3	Al_2O_3	$5CaO.3Al_2O_3$	$3CaO.Al_2O_3$	$2CaO.SiO_2$	$3CaO.SiO_2$
		$2CaO.SiO_2$	$2CaO.SiO_2$	$3CaO.SiO_2$	$3CaO.Al_2O_3$
SiO_2	SiO_2	$3CaO.Al_2O_3$	$3CaO.SiO_2$	Eutectic	

In reality, on account of the presence of small quantities of oxide of iron, of magnesia, alkalies, etc., the temperature obtaining inside the furnace and the constitution are slightly different. In practice, according to the nature of the cement manufactured, the following results are obtained for a well heated and treated cement (Table 2).

If the cement has not been properly heated, a portion of the lime and the bisilicate of calcium which should have entered into a combination to form a trisilicate have not come to react with each other, with the consequence that bisilicate subsists, and its presence in the beta modification in the cement as has been shown, is causative of the cement's destruction by swelling and pulverization. The mechanism of hardening of set cement is not yet well understood, but the mechanism of the setting is known almost certainly. These are the phenomena that happen when pulverized cement is tempered with water. Pure trialuminate of calcium, $3CaO.Al_2O_3$, like pure trisilicate of calcium, $3CaO.SiO_2$, set rapidly and then harden very fast, but the aluminate sets appre-

TABLE 2.

Type.	Composition of raw materials.				Maximun heating.	Composition of clinker.
Chemically pure cement.....	CaO.....	68.2		100	1650°	$2CaO.SiO_2$ $3CaO.SiO_2$ $3CaO.Al_2O_3$
	Al_2O_3.....	8.2				
	SiO_2.....	23.6				
White cement.....	CaO.....	66.2	97.6	100	1525°	$2CaO.SiO_2$ $3CaO.SiO_2$ $3CaO.Al_2O_3$ CaO in small quantities
	Al_2O_3.....	6.4				
	SiO_2.....	25.0				
	MgO, Fe_2O_3, Na_2O. K_2O.....	.....	2.4			
Gray cement (ordinary grade)	CaO.....	63.2	93.3.	100	1425°	$2CaO.SiO_2$ $3CaO.SiO_2$ $3CaO.Al_2O_3$ $CaO.3Al_2O_3$ in small quantities CaO and ferrite
	Al_2O_3.....	7.7				
	SiO_2.....	22.4				
	MgO, Fe_2O_3, Na_2O K_2O, SO_3.....	.....	6.7			

ciably quicker than the silicate. Both of them form, first, amorphous hydrates with water. The bisilicate of calcium, $2CaO.SiO_2$, on the contrary, reacts very slowly with water. Not only is setting slow, but also its hardening requires very much time. Then also the free lime fixes water and gives rise in its turn to a hydrate crystallizing at the same time as this occurs with the amorphous hydrate formed by trialuminate of calcium. It does not seem that the hydrates formed by the bisilicates and trisilicates of calcium crystallize. Hence, the setting is due above all to the hydration of the aluminate, the hardening above all due to the hydration of the aluminate which continues, and to that of the trisilicate. The trisilicate, as a consequence, is that constituent of the clinker which contributes most to the cementation, seeing that the bisilicate sets and hardens at too slow a rate and the aluminate too rapidly. Moreover, the latter is rather soluble in water and it never becomes very hard. Hence, a cement is so much superior in quality, the higher the quantity of trisilicate of calcium it encloses. Now, the temperature at which it forms from lime and silica lies in the neighborhood of 1700°, which is much higher than the temperature ordinarily reached in furnaces, whence there results the necessity of having recourse to a flux facilitating its formation. Now, it is especially aluminate of lime which plays the part of the flux in the last phases or stages of the heating. The investigation of CaO-Fe_2O_3 made by Rankin demonstrates that Fe_2O_3 is able to play this part better than Al_2O_3 and may replace it in part. By further following up the investigation of this system, one may perhaps succeed in finding bases which may permit of obtaining products of definite properties in the fabrication of ferro-concrete (Eisenportlandzement) in which the Germans have been specializing. C. TREISCHEL.

Setting of plaster. C. H. DESCH. *Trans. Ceram. Soc. (England)*, **18**, Part 1 (1918).—LeChatelier has shown that gypsum, $CaSO_4.2H_2O$, passes, at about 128°C, into the hemihydrate, $2\ CaSO_4$. The bulk of ordinary plaster is hemihydrate which is more soluble than the hydrate. On mixing with H_2O a solution, saturated with respect to the hemihydrate and supersaturated with respect to the dihydrate, results. Crystallization of gypsum takes place spontaneously or around nuclei furnished by the plaster itself. The strength of set plaster is attributed to interlocking lath-shaped crystals. Volume changes in setting are attributed to crystals pushing each other apart during growth. Effect of overburning on setting of plaster is also discussed. The general discussion is interesting. C. TREISCHEL.

CERAMIC PATENTS.

G. E. MIDDLETON, *Abstractor.*

General.

Water-purifying material and process of making it. H. KRIEGSCHEIM. U. S. 1,312,552, Aug. 12, 1919. In softening water it is the practice to use base-exchanging material containing in combination SiO_2, Al_2O_3, alkalies and

water–zeolites. The water is passed through a bed of this material exchanging the lime and magnesia, to which it owes its hardness, for the sodium contained in the base-exchange material. After a sufficient degree of action is attained the material may be revived by treatment with NaCl. The revived silicate is then ready for re-use. It is necessary that these base-exchange materials be mechanically strong and able to resist the action of water, a property which they do not naturally possess. The inventor claims that they may be heated to as high as 550° C for not more than two hours without substantial or permanent loss of the water of constitution upon which their activity depends. He therefore incorporates them in finely divided condition with a small proportion of clay and subjects the product to the above heat treatment. A hard, rigid but active material results which may be formed into pellets. Decayed obsidian is instanced as an example of the base-exchange material.

Clay-binder and process of making it. H. L. KOHLER. U. S. 1,312,853, August 12, 1919. Non-plastic clays, notably flint clays, are treated by this process to increase their bonding power and plasticity and also to enable them to be fired without danger of cracking. These results are said to be attained by adding a basic salt of aluminium to, or forming it within the clay. The basic salt operates as a binder during tempering and molding and during firing is gradually converted to its oxide without losing its effectiveness as a binder. From $^1/_2\%$ to 5% of anhydrous $Al_2(SO_4)_3$ is thoroughly mixed with wet clay and allowed to remain at atmospheric temperature. The reaction represented by the following is said to take place: $3(Al_2O_3.2SiO_2)+2Al_2(SO_4)_3+xH_2O = 4Al_2SO_4(OH)_4+Al_2(SO_4)_2(OH)_2+6H_2SiO_3+xH_2O$. In the firing these basic sulphates are converted into oxides and a portion of the latter are converted into silicates either by the silicic acid set free in the above reaction or by said acid and any free silica which may be present in the clay. At red heat the SO_3 radical is driven off and Al_2O_3 remains, a portion of which reacts with the silicic acid or free silica as follows: $Al_2O_3+H_2SiO_3+SiO_2=Al_2O_3.2SiO_2+H_2O$. The use of $Al_2(SO_4)_3$ is not imperative. Any mineral acid or salt capable of setting free the silicic acid of the aluminium silicate of the clay may be used. H_2SO_4, $(NH_4)_2SO_4$ and even $Zr(SO_4)_2$ are mentioned. Other argillaceous materials such as bauxite may be similarly treated and with similar results.

Means for supporting ceramic ware while being fired. H. J. RUSHTON. U. S. 1,313,018, Aug. 12, 1919. Several types of supports are disclosed, all being of open structure and designed for use in muffle kilns. One type comprises a rectangular base and end walls, the base being provided on its lower side at one end with a central foot or lug and at the other with a pair of laterally arranged feet or lugs designed to coöperate with members on the top of the end walls of a companion member, the whole arrangement to permit of sliding due to expansion of the parts when the supports are fired in superposed relation.

Tile-cutting machine. C. A. Thorpe. U. S. 1,313,213, Aug. 12, 1919. The movement of the extruded hollow-tile bar governs the movement of the cutting table carrier. The carrier follows a curved path adjustable to correspond to any curvature which it may be desired that the tile possess. Automatic means are provided for cutting the tile radially.

Brick-mold. H. W. B. Graham. U. S. 1,314,057, Aug. 26, 1919. A multiple brick mold designed for use in the Graham soft-mud brick machine. The mold is provided at its ends with supporting members extending laterally beyond the bottom boards and provided with upper and lower wide flat-bearing surfaces. These supporting members are adapted to engage with various parts of the brick machine as the mold moves through it.

Paving-brick. W. J. McClain, Jr. U. S. 1,314,278, Aug. 26, 1919. This paving brick is designed to be laid flat instead of edgewise. It is provided with lugs on its lateral edges for interlocking with lugs on other bricks, these lugs being so located that they not only serve to space the brick from those bricks lying laterally thereto and interlocked therewith, and at the same time provide intermediate lateral spaces for the reception of joint-filling material, but they also serve to space the brick endwise from adjacent bricks.

Kiln. H. Francart. U. S. 1,314,486, Aug. 26, 1919. Checker-work furnaces extending from the crown to the floor are longitudinally disposed along the sides of this kiln, and communicate with the kiln chamber at their top and bottom. Air and gas are introduced from beneath the bottom of each furnace. The exhaust flue is located near the floor at one end. The inventor states that the products of combustion rise in each furnace, enter the kiln chamber at the crown, impart their heat to the ware and are drawn into the next furnace toward the exhaust flue at the floor level, thus pursuing a helicoidal path. This together with the heat stored in the checker work makes for uniformity of temperature.

Article adapted to be used as a light-reflector. H. Ford. U. S. 1,313,806, Aug 19, 1919. Pieces of alabaster or gypsum are imbedded in Keene's, Parian or Martin's cement. These cements are translucent and so the mixture is adapted to be made in shades or light-reflectors.

Mold for ceramic products. F. S. Lowry. U. S. 1,314,821, Sept. 2, 1919. This mold is designed to provide offset handle-receiving projections on ordinary jiggered cooking utensils. Formerly these projecting ears have been formed by appropriate depressions in the mold wall adjacent to its upper periphery, but this resulted in making the ears flush with the upper edge of the utensil. In order to lower them the inventor provides the mold with removable inserts which fit over the ear molds.

Clay-pin drying and cutting machine. D. D. Irwin. U. S. 1,315,507, Sept. 9, 1919. So-called clay-pins used to space articles of pottery apart during firing are dried and cut into appropriate lengths by this machine.

Pallet boards containing strips of die-expressed pin material are passed upon an endless conveyor through a steam-heated drier. After the clay has dried to approximately a leather-hard condition the strips are passed under a gang of rotary disc cutters and cut to length. The cut pins then proceed through the remaining length of the drier and are discharged in condition for firing.

Brick-veneering machine. J. ELDRIDGE. U. S. 1,315,916, Sept. 9, 1919. Brick are automatically fed to a conveyor and carried beneath coating mechanism to receive a coating of slip or glaze. After coating the brick are wiped at the corners leaving them square. More than one face of the brick may be coated.

Abrasives.

Abrasive material and method of making it. N. C. HARRISON. U. S. 1,314,061, Aug. 26, 1919. Diaspore is calcined and thoroughly mixed with previously ground coke and iron. Waste iron, such as cast iron borings, is suitable. The mixture is then fused in an electric furnace and allowed to cool and crystallize slowly. The finished product is said to be a compact, fine-grained mass, free from pores, making an excellent abrasive material, harder and tougher than corundum. The proportions of ingredients used to make a finished ton of abrasive are: diaspore, 3400 lbs., iron borings, 400 lbs., and coke, 600 lbs.

Manufacture of artificial abrasives. A. T. SNODGRASS. U. S. 1,314,835, Sept. 2, 1919. "Flour" emery, the finely powdered waste product from emery and glass-grinding mills, is used as the basis for this abrasive. To this is added another waste product, "flour" glass, the fine residue obtained from grinding cullet in the manufacture of glass paper. The proportions are 2 to 3 parts of emery to 1 part of glass. The two are intimately mixed and subjected to a temperature of not less than 1700°C. The resultant molten mass is poured on to a cool surface or otherwise allowed to solidify. When crushed and graded it is said to be suitable as a raw material in the manufacture of abrasive cloth, paper, and grinding wheels.

Apparatus for the fusion of refractory materials. R. A. HOYLE. U. S. 1,314,871, Sept. 2, 1919. This apparatus is especially designed for the fusion of bauxite in the manufacture of artificial abrasives, the inventor seeking to simplify the complicated furnace installations now employed for that purpose. A series of pits are arranged in rows on the mill floors, each constituting the crucible of an electric furnace. The electrodes are suspended from a traveler moving on overhead trackways so arranged that they may be lowered into any pit of the series. A hood adapted to cover a pit is similarly suspended. In operation the electrodes are lowered in the first pit, the hood fixed in place and the bauxite gradually fed in. As the ore melts the electrodes are raised. The material adjacent to the walls of the pits is not subjected to the extreme heat, is not fused and serves as a protection for these walls. When fusion is complete the electrodes are shifted to the adjacent pit. The cooled melts

are withdrawn by means of chains laid along the sides and bottom of the pit before introduction of the charge.

Glazes and Enamels.

Process of making opaquing- and enameling-compounds and the product. F. PREUSSER. U. S. 1,314,831, Sept. 2, 1919. The object of this invention is to reduce the quantity of the expensive opacifying oxides such as those of tin, zirconium and antimony used in glazes and enamels and at the same time effect the same degree of opaqueness. An opacifier meeting these requirements is said to be obtained by the mutual decomposition and reaction of two groups of enameling substances, one of these being the turbidity producing or opaquing-salts and compounds of the alkaline earth metals and some earth metals, the oxides of which do not possess this quality, while the other group consists of the opaquing oxides or compounds of the so-called border elements, that is those mostly tetravalent and partly trivalent comparatively, easily fusible, metallic elements which partake of the characteristics of metals as well as metalloids, such for instance as tin, titanium, zirconium, thorium, antimony and to some extent bismuth, arsenic and cerium. These two groups must be chemically combined. This combination may be effected in either the dry or the wet way. Phosphates, silicates and aluminates of the latter-mentioned metals which form opaquing oxides and chemical combinations thereof with the alkali earth metals are thus produced in which, owing to the mutual decomposition which has taken place, the clouding oxides such as SiO_2 are contained in an exceedingly fine, almost colloidal distribution, similar to a dyeing agent, so that this artificial product and the opaquing and enameling results thereby produced are entirely different from the natural silicates of the rare earth metals and the very weak clouding effects produced by them.

Several examples are given. One follows: Proceeding in the wet way, $ZrOCl_2$ 55 parts, $MgSO_4$ 2.5 parts, and $K_2Al_2(SO_4)_4.24H_2O$ (potash alum) 5 parts, are dissolved in water and treated with a solution of Na_3PO_4 20 parts, Na_4SiO_4 5 parts and NaOH 10 parts. A complex body containing silicates and phosphates of zirconium and aluminium with some phosphate and silicate of magnesium is formed. The soluble and volatile decomposition products are washed out. Proceeding in the dry way $Zr(OH)_2$, Al_2O_3 or $Al(OH)_3$ and $Ca_3(PO_4)2$ are mixed and melted, and, if necessary, a small amount of $MgCO_3$ or $Na_2B_4O_7$ may be added to assist in the decomposition of the phosphate as well as for the purpose of regulating the melting of the mass and the adhering quality of the enamel.

The inventor states that these compounds may be used alone as glazes or in combination with ordinary fritted glazes as opacifiers.

Clouding-composition for enamels and process of making it. P. EYER. U. S. 1,314,861, Sept. 2, 1919. ZrO_2 and other zirconium compounds have

been substituted for SnO_2 as opacifiers but they produce spotted and dull or tarnished enamels. Zirconium borate is comparatively free from these defects but its covering capacity is insignificant. The inventor has found that zirconium borate strongly supersaturated with zirconium oxide, such for instance as a product of the composition $ZrO_2.2B_2O_3 + 4ZrO_2$, has a greater covering power than ordinary zirconium borate and at the same time does not cause spotting or dullness. The covering power of the compound is also increased by melting cryolite with it. Cheap, raw zirconia can be used provided an oxidizing agent is employed. Example: Raw ZrO_2 70 parts, B_2O_3 30 parts, Na_3AlF_6 20 parts, $NaNO_3$ 50 parts, are thoroughly mixed, fused and ground very fine.

Refractories.

Method of manufacturing magnesite refractories. R. D. Pike. U. S. 1,312,871, Aug. 12, 1919. California magnesite runs higher in MgO than the Austrian, but the physical properties of bricks made from the latter, calcined, are much superior. Austrian magnesite bricks, fired to cone 18, are hard, dense and strong while those made from the California material are soft and have little strength. If the physical properties of the American product could be improved it would be a superior basic refractory because of its high MgO content. The inventor adds to this magnesite from 0.32% to 0.75% of powdered ferro manganese and from 1.5% to 2.5% of ferric oxide in the form of powdered iron ore or mill scale as low in impurities as possible. The whole is ground in a wet pan, molded into blocks and fired to cone 18. The iron oxide and ferro manganese react as follows: $8Fe_2O_3 + FeMn_4 + heat = 16FeO + Fe + 4MnO_4$. The ferrous and metallic iron are active fluxes or bonding agents. A hard, dense, strong magnesite refractory containing high percentages of MgO is said to result.

Glass.

Manufacture of glass. H. A. Galt. U. S. 1,312,784, Aug. 12, 1919. Niter cake, a by-product in the manufacture of HNO_3 from $NaNO_3$, and cylinder cake, a by-product in the manufacture of HCl from NaCl, consist of a relatively large proportion of $NaHSO_4$ with a relatively smaller proportion of Na_2SO_4. It has been considered impractical to use them as ingredients of a glass batch because of their deliquescence and acidity, resulting in the formation of "salt water" which lies on the surface of the molten batch, attacks the container, interferes with the melting and handling and produces a scum on the glass. These objectionable features are obviated by grinding the niter or cylinder cake with a neutralizing base and adding it to the batch. As examples of this neutralizing base, Na_2CO_3 (soda ash), $NaHCO_3$, $CaCO_3$ (limestone) and $Ca(OH)_2$ are given. The following is a typical batch formula:

Sand	1000
Soda ash	298
Mixture:	
Soda ash 16 / Niter cake 50	66
Salt (NaCl)	25
Limestone	309
Coal	3
Arsenic (As_2O_3)	5

Apparatus for feeding and delivering glass. J. SCHIES. U. S. 1,312,876, Aug. 12, 1919. A series of molds upon a rotary mold-table are successively passed beneath the glass feed. As each mold receives the required quantity of molten glass the flowing glass stream is automatically severed and deflected.

Lens-forming process. A. PELLOW. U. S. 1,312,920, Aug. 12, 1919. In forming a fused bi-focal lense a spherical countersink is formed in the major blank and a segment having a contrageneric toric surface is placed in contact with it. One of the curves of the segment is less and the other greater than that of the countersink. The two parts are then subjected to heat which softens this segment and causes it to roll out and assume the curvature of the countersink, forcing out all air bubbles in advance of contact between the parts.

Apparatus for rolling ribbed glass. W. COX. U. S. 1,313,071, Aug. 12, 1919. The cold spot and consequent peacock-feather appearance of ribbed glass are obviated by the use of a rolling table having shallow wide-angled grooves with flat-faced ribs between them cut in the surface of the table, about 20 grooves to the inch. The angle of the flat sides of the ribs between the grooves is from 5° to 40° to the plane of the table.

Glass-blowing apparatus. H. E. and C. L. QUACKENBUSH. U. S. 1,313,205, Aug. 12, 1919. This machine is especially adapted for blowing incandescent lamp bulbs. A glass tube is rotated without being subjected to a torsional strain so that when the glass is molten at an intermediate point in its length it will be sustained at such point without torque. Thus the tube can be successively treated at points along its length and converted into a series of bulbs arranged end to end.

Apparatus for annealing glass. J. W. CRUIKSHANK. U. S. 1,313,222, Aug. 12, 1919. Difficulty is experienced in cooling sheets of glass sufficiently during annealing so that when they are removed from the cool end of the lehr they will not crack upon contact with the cold air, especially in winter. Lengthening the lehr is impracticable. The inventor cools by convection, allowing access of air to the sheets at the lower end by increasing the size of the lehr runway or cooling chamber to the full size of the building containing

it. A general circulation of air is thus attained and the air is cooled by contact with the walls and ceiling of the surrounding building.

Apparatus for building up objects of quartz glass. W. S. QUIMBY and F. W. ROBINSON. U. S. 1,314,212, Aug. 26, 1919. Heretofore articles of quartz glass have been produced by merely drawing out a block of quartz glass heated to a plastic condition. This is expensive and involves much skill. The inventors build up objects by applying quartz powder to a heated nucleus of quartz glass and reheating to fuse the accretion. If the object to be made is a tube, the initial nucleus is made tubular and held in shape during the operation by compressed air within it. The nucleus tube is rotated by two supporting chucks and heated to plasticity by a gas burner. The burner is removed and a plate covered with quartz powder is moved beneath and in contact with the tube. The powder adheres and is then made to fuse onto the tube upon reheating with the burner. At the same time the work is elongated by drawing its ends apart sufficient to compensate for the added bulk of powder. This operation is then repeated until the desired length is reached.

Apparatus for making glass cylinders. G. C. DEBAY. U. S. 1,314,638, Sept. 2, 1919. A hydraulically operated apparatus for drawing glass cylinders. The cylinders are drawn through rings placed on the surface of the molten glass. When a cylinder has been drawn its full length these rings are raised whereupon the heat of the furnace melts the glass at the lower end and disconnects the cylinder from the glass in the tank. Hot air is admitted to the cylinders during drawing. The apparatus is designed to draw two cylinders simultaneously.

Mechanism for delivering glass. R. NETTER. U. S., 1314,691, Sept. 2, 1919. Molten glass is fed to molds situated on a continuously rotating mold table. An auxiliary spout follows each mold through a predetermined arc of its travel; the flow of glass is then automatically shut off and the spout returned to contact with the mold next succeeding.

Mechanism for effecting spindle movements in glass-forming apparatus. A. KADOW. U. S. 1,315,130, Sept. 2, 1919. This machine is particularly designed for the manufacture of electric-light bulbs or other hollow-glass articles. The blank is developed on a spindle or blow-pipe by blowing, by swinging the blank out through oscillating movements of the spindle and by revolving the spindle on its longitudinal axis with the blank inclosed in the mold. In order to fix or set the blank in the holding jaws of the spindle and center the air employed in blowing, the spindle is provided with a plunger which is thrust into the inner end of the blank immediately after the blank has been received from the gathering mechanism. Improved mechanism is provided for operating the plunger without sidethrust. Improved means are also provided for accurately centering the spindle.

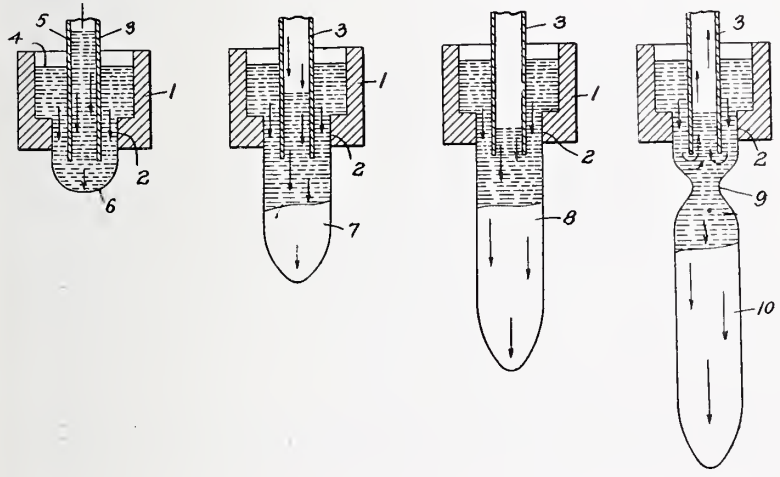
Charging apparatus for glass-molds. R. T. MCGEE. U. S. 1,315,562, Sept. 9, 1919. Glass is fed to a press mold without being exposed to the air

or the chilling which results from the use of a relatively cold ladle or other gathering instrument. The glass flows horizontally from the bottom of the tank to the bottom of a vertical well at the top of which the mold is seated. Extending laterally from the bottom of the well and in direct communication with the horizontal duct is another horizontal passageway leading to an adjacent vertical, cylindrical, auxiliary tank at the top of which is a water-cooled, loosely fitting plunger. All of the ducts mentioned and the lower part of the auxiliary tank are surrounded by highly heated fire spaces to prevent chilling of the glass therein. The glass rises in the well because of the pressure in the tank and is forced into the bottom of the awaiting mold by depressing of the plunger in the auxiliary tank. As the mold is filled it is slid transversely, shearing the glass column and making way for a succeeding mold. In a modified form the glass flows out at the top of the well and drops into an open-topped mold.

Method of feeding glass. G. E. HOWARD. U. S. 1,315,668, Sept. 9, 1919. The diameter of a stream of molten glass flowing through an orifice may be varied by varying the pressure which is forcing the glass therethrough. This is due to the fact that the adherence to the edge of the orifice is greater than the internal friction of the molten glass. Thus it is possible by varying the flow to expand or contract the section of the stream near the orifice and make it assume a predetermined form within narrow limits. Advantage is taken of this fact in forming gobs of desired size for discharge to a mold.

The glass flowing down through the orifice first assumes the more or less globular shape 6 shown in Fig. 1. At the same time that this initial flow takes place, a certain amount of glass may be drawn up into the tube 3, then as the flow continues out through the orifice 2 and when the globule 6 elongates,

FIG. 1 FIG. 2 FIG. 3 FIG. 4



and thereby tends to become attenuated or thin, the glass in the tube 3 may be forced out as shown in Fig. 2 to fill in the center of the globule and cause the globule to assume substantially the form shown at 7, Fig. 2. Continued expulsion of the glass from the pipe 3, as illustrated in Fig. 3, causes the globule to maintain substantially a uniform diameter, as shown at 8, Fig. 3, so that there then is a mass of glass ready for discharge into a mold, or the like, which is of substantially uniform diameter and practically predetermined shape, and mass.

When now it is desired to cut off this globule to leave the same in the mold, the sucking action into the tube 3 may be resumed, as shown in Fig. 4, and may be made sufficiently rapid to cause a decided contraction in the globule at the point 9. It will be understood, however, that the flow of the glass down between the orifice 2 and the walls of the pipe 3 has not thus been checked, but after the glass has passed into and partially out of the orifice it is drawn up into the tube 3 instead of being supplied to the completed globule 10. The glass globule is then cut off at the point 9 in the ordinary manner.

Method and apparatus for manufacturing incandescent-lamp bulbs. R. Le Rossignol. U. S. 1,315,783, Sept. 9, 1919. A tubulator through which an incandescent-lamp bulb may be exhausted is formed in the wall of the stem or flare and not in the bulb itself by locally heating the desired spot on the stem and inserting a mandrel in the thus softened glass.

Bulb-cutting machine. R. W. Suman. U. S. 1,315,799, Sept. 9, 1919. A machine for automatically feeding and cutting the necks of miniature lamp-bulbs blown from glass tubing.

Method of manufacturing incandescent lamps. M. H. Branin. U. S. 1,315,813, Sept. 9, 1919. Incandescent-lamp bulbs are blown with elongated unrestricted necks terminating in a heavy ring of glass. The filament support may be readily inserted in such a neck without first trimming it as has heretofore been necessary with bulbs having necks with restricted ends. The final sealing may be effected as usual.

Cement.

Manufacture of potash and cement. A. C. Spencer. U. S. 1,312,592, Aug. 12, 1919. Cement is made using potash-bearing minerals such as orthoclase feldspar, glauconite, nephelite, mica etc., and the potash recovered in water-soluble form. The process is characterized by two clinkering operations and the use of either an excess or a deficiency of lime in the first clinkering. The clinker obtained by the first operation is pulverized preferably by the use of water. The potash removed in the two clinkering operat ons is collected from the dust and the gases of the kiln in any suitable way. The inventor claims a higher yield of potash by this method than by those described in U. S. 1,209,135 and 1,209,219.

Rotary-kiln. J. S. Fasting. U. S. 1,313,281, Aug. 19, 1919. The feeding-end of this rotary cement kiln is provided with a series of helically arranged

chains secured to the inner wall of the kiln with the inner end of each chain in advance of the outer end circumferentially and in the direction of rotation of the kiln. As the kiln rotates the chains lift the slurry, exposing it to the action of the furnace gases and at the same time, because of their helical arrangement, cause it to pass toward the discharge end of the kiln.

Process of making porous concrete. E. WALTER. U. S. 1,313,931, Aug. 26, 1919. Small pellets of paraffin are mixed with cement mortar. When the resulting concrete has set the paraffin is removed by melting it or by extracting it with solvents such as gasoline, benzine or carbon tetrachloride. A porous concrete is said to result.

Rotary kiln. P. A. BOECK. U. S. 1,314,461, Aug. 26, 1919. In the lower temperature zones of this rotary cement-kiln a layer of kieselguhr bricks is interposed between the refractory lining of the kiln and the outer shell. The kieselguhr tends to prevent heat losses and the result is a higher temperature in the lower temperature zones.

Burned shale and method of preparing it, and concrete made therefrom. O. OLSEN. U. S. 1,314,752, Sept. 2, 1919. Shale rock is fired in a kiln at a temperature of 2000° F or above, for a period determined by the nature of the rock, so that it is reduced to a light, porous, hard material. It is stated that this product is useful in the refining of sugar or oil but the inventor discusses its use primarily as an aggregate for concrete. The following concrete mixture is given: coarsely crushed, burned shale 4 parts, finely crushed burned shale 2 parts, portland cement plus 10% magnesia cement 1 part. Concrete made according to this formula is said to weigh from 80 to 84 pounds per cubic foot, to resist a temperature of 3800° F without injury and to possess great tensile and compressive strength. It is especially adapted for marine work, barges, ships and railroad ties.

Composite building material. E. VIENS. U. S. 1,314,772, Sept. 2, 1919. The material has the following composition: Portland cement 30–65%, asbestos or other non-combustible fiber 10–55%, hydrated lime 2–10%, siliceous material 2 to 15%, sawdust or equivalent 2 to 10%. It is said to be fireproof and resilient and above all possesses the property of taking nails, tacks or screws even better than wood.

Process and apparatus for cooling cement-kiln gases and the recovery of dust therefrom. L. STEVENS. U. S. 1,315,254, Sept. 9, 1919. Cement-kiln gases carrying suspended matter are mingled with a regulated amount of water-saturated air immediately upon their exit from the kiln and then further mingled with a current of cool air. The gases thus cooled are passed through a dust separator and the suspended matter precipitated in a physically dry condition.

Plaster composition. L. V. ECKERT. U. S. 1,315,597, Sept. 9, 1919. A plaster composition composed of cement plaster and straw meal in substantially equal proportions.

Note.— Copies of these patents may be obtained for ten cents each by addressing the "Commissioner of Patents, Washington, D. C."

AMERICAN CERAMIC SOCIETY.

Acquisition of New Members during October, 1919.

Resident Associate.

Bachman, P. S., 92 W. Maynard Ave., Columbus, Ohio.
Brown, Byron P., Vice-president and Gen. Mgr., Hercules Porcelain Co., Belvidere, Ill.
Charron, Roy C., Supt. and Chemist, Crunden Martin Mfg. Co., St. Louis, Mo.
Chu, Kea Hin, International General Electric Co., Harrison, N. J.
Flint, Francis C., Chief Chemist, Hazel-Atlas Glass Co., Washington, Pa.
Hollmayer, John G., Sec'y and Treas., K. B. H. American China Co., 5210 Norwood Park Ave., Chicago, Ill.
Jones, Chester H., Industrial Editor "Chemical and Metallurgical Engineering," 1570 Old Colony Bldg., Chicago, Ill.
Kahn, Isaac, Owner, Wheatley Pottery Co., 2428 Reading Rd., Cincinnati, Ohio.
Kenyon, S. Spicer, Alfred University, Alfred, N. Y.
Krebs, A. E., Belleville Enameling Works, So. 16th St., Belleville, Ill.
Ling, S. C., International General Electric Co., Harrison, N. J.
Panchot, L. D., 2nd Asst. Supt., Pittsburgh Plate Glass Co., Ford City, Pa.
Proodian, E. K., Chemist and Asst. Supt., Florentine Pottery Co., Cambridge, Ohio.
Rahn, Robert C., Ceramic Engineer, Western Electric Co., 4642 West End Ave., Chicago, Ill.
Reid, William H., Alfred University, Alfred, N. Y.
Sheppard, Mark, Fellow in Refractories, Mellon Institute, Pittsburgh, Pa.
Sherwood, Robert F., Ceramic Assistant, Bureau of Standards, Pittsburgh, Pa.
Smyser, Albert E., Engineer of Refractories, 239 South Fairmont St., E. E., Pittsburgh, Pa.
Talbert, E. Hume, Patent Lawyer, 711 Eighth St., N. W., Washington, D. C.
Wilkins, Wm. W., 2608 Leland Ave., Chicago, Ill.

Foreign Associate.

Catecliff, John, Chemist, Lodge Sparking Plug Co., Ltd., Rugby, England.

ACTIVITIES OF THE SOCIETY.

Actions of the Board of Trustees:

Nov. 24th. President R. T. Stull appointed Mr. H. F. Staley as delegate from the American Ceramic Society to the Industrial Safety Codes Conference, at the Bureau of Standards, Washington, December 8, 1919.

Nov. 26th. It was voted to sanction the purchase of an adding machine for the Secretary's office.

Nov. 26th. It was voted that all copies of the Index to the Transactions be bound in uniform manner with the bound volumes of the Transactions.

Nov. 26th. It was voted to send the Journal of the Society to the Association de Documentation Bibliographique, Scientifique, Industrielle, et Commerciale, of Paris, France, in return for a place, with critical notice, for the Journal in an Index which the Association is to publish.

Nov. 26th. It was voted to elevate Professor O. J. Whittemore, of Iowa State College, Ames, Iowa, to active membership.

LOCAL SECTIONS.

Northern Ohio Section.

HOTEL CLEVELAND, CLEVELAND, OHIO.

DEC. 1, 1919.

The following program will be afforded:

1.30 P.M. CERAMIC SYMPOSIUM:

1. "Testing Fire Bricks," by Dr. A. F. Gorton, Ceramist, Buckeye Clay Pot Co., Toledo, Ohio.
2. General discussion on refractories.
3. General discussion on fuels.
4. General discussion on ceramic problems.

Business Session. Report of Secreatry-Treasurer for 1919. Installation of Officers.

B. A. RICE,
Secretary.

New York State Section.

ALFRED UNIVERSITY, ALFRED, N. Y.

DECEMBER 15 TO 17, 1919.

A short course in ceramics will be held in connection with this meeting. The object of the short course is to give managers,

superintendents, foremen and workmen in ceramic plants an opportunity to gather in a few days a general understanding of the technology of the industry in which they are interested. Ample opportunity will be given for discussion of any subject which may be raised so that any one having problems which are giving trouble will be greatly benefited by attending the course. The program follows:

MONDAY, 1.30 P.M.

Lecture: "Developments and Prospects in Ceramics," by C. F. Binns, Alfred University, Alfred, N. Y.

Laboratory demonstration of deflocculation and elutriation, by C. F. Binns.

Laboratory demonstration by J. B. Shaw and G. A. Bole, Alfred University, Alfred, N. Y. Califoric value of coal; Fusion point of ash; Specific heat of ceramic products.

8.00 P.M.

Reception by the Faculty at the home of Professor and Mrs. Binns.

TUESDAY, 9.00 A.M.

Lecture: "The Burning Process," by A. V. Bleininger, Bureau of Standards, Pittsburgh, Pa.

Laboratory demonstration by J. B. Shaw and G. A. Bole: Heat balance of a kiln; Burning behavior of a clay.

1.30 P.M.

Lecture: "Porcelain," by A. V. Bleininger.

Laboratory demonstration by J. B. Shaw and G. A. Bole: Slag test of fire brick; Spalling test.

Calibration of electric pyrometers, by W. A. Titsworth, Alfred University, Alfred, N. Y.

8.00 P.M.

Studio demonstration: Making and decoration of pottery, by Elsie Binns and Marion Fosdick, Alfred University, Alfred, N. Y.

Reception by Department of Applied Art.

WEDNESDAY, 9.00 A.M.

"The Proctor Dryer," by D. H. Applegate, Philadelphia Textile Machinery Co., Philadelphia, Pa.

"The Tunnel Kiln," by P. Dressler, American Dressler Tunnel Kiln Co., New York City, N. Y.

1.00 P.M.

"Refractories for the Glass Industry" by A. V. Bleininger.

"Special Refractories," by S. C. Linbarger, Carborundum Co., Niagara Falls, N. Y.

"Electrical Porcelain," by L. E. Barringer, General Electric Co., Schenectady, N. Y.

J. B. SHAW,
Secretary.

Eastern Section.

RUTGERS COLLEGE, NEW BRUNSWICK, N. J.
DEC. 18, 1919.

The winter meeting of the New Jersey Clay Workers' Association and Eastern Section of the American Ceramic Society will be held as usual at Rutgers College, New Brunswick, N. J. The following program will be afforded:

MORNING SESSION: 10.30 A.M.

Address: By Charles Howell Cook, Chairman.

"Properties of Some Pottery Bodies and Glazes," by H. G. Schurecht, Bureau of Mines, Columbus, Ohio.

"Feldspar as a Pottery Material," by H. F. Staley, Bureau of Standards, Washington, D. C.

LUNCHEON: 1.00 P.M.

AFTERNOON SESSION: 2.00 P.M.

"The New Jersey Museum of Ceramic Products," by Helen C. Perry, State Museum, Trenton, N. J.

"Fuel Analyses," by A. C. Fieldner, Bureau of Mines, Pittsburgh, Pa.

"The Use of Fuel Oil in the Firing of Ceramic Kilns," by R. L. Clare, Federal Terra Cotta Co., Woodbridge, N. J.

"A Producer-gas Fire Box," by R. H. Minton, General Ceramics Co., Metuchen, N. J.

G. H. BROWN,
Secretary.

INDUSTRIAL DIVISIONS.

The following letter has recently been forwarded to all officers of the Industrial Divisions of the Society.

DEAR SIRS:

The membership work has been decidedly neglected the last month or so by the Membership Committee as a body, although each one has been doing

some individual work. The bulk of the work which has been done in securing new members has been by the Divisions and if they function as they should the Society may look to them to secure a great many members with whom no one else would come in contact. Particularly commendable work has been done by the Glass Division, the Terra Cotta Division, the Refractories Division, and the Enamel Division. Some of the Divisions have shown no activities whatever and it is hoped that they will arrange programs in somewhat similar manner to the other Divisions so that they may have their share of prominence in the Society.

As you know, the Board of Trustees voted that ten per cent of the dues and fees, as of 1919, of members who have, or shall have, become members of an Industrial Division, shall be allotted to that Division for legitimate expenses such as circulars, letters, postage, stenographic expenses, etc. This should not only assist in the work of your Division but will mean that each new member you secure will strengthen your particular Division, not only in numbers, but also financially. It is hoped that all of your members will work with this fact in view as we feel sure it will greatly assist in increasing the membership of the Society as a whole.

One of the greatest things the Divisions can do to keep alive the enthusiasm of the members would be to arrange a publication committee or in some other way have your Division coöperate with the Editor and Publication Committee so that an article of interest to your Division would appear in each issue of the JOURNAL.

You will note in the September issue that the Glass Division not only has an article on glass but also has notes of interest to the Glass Division. Also note that this Division has arranged for and appointed a Central Committee, not only to create enthusiasm, but also to answer questions concerning everyday glass practice. Undertakings of this sort cannot help but increase the strength of their Division.

The Terra Cotta Division has outlined eleven lines of research upon which work is being done throughout the country. They will, no doubt, present papers on these subjects at the coming meeting and will also have one of these papers published in each of the issues of the JOURNAL, meaning, of course, that something of interest to the Terra Cotta Division will be in each JOURNAL.

It is earnestly hoped that all of the Divisions will create as much enthusiasm among their members as the two Divisions just mentioned. The Membership Committee will do all it can to assist in this work and will greatly appreciate any suggestions or advice.

It would be helpful to the Membership Committee if the secretary of each Division keep the writer posted as to the Division activities.

Yours very truly,

F. H. RIDDLE,
Chairman, Membership Committee.

AMERICAN CERAMIC SOCIETY.

Annual Meeting.

The annual meeting of the Society will be held at the Bellevue-Stratford Hotel, Philadelphia, Pa., February 23 to 26, 1920.

At the call of President Stull, eighteen members met in Philadelphia on the evening of Wednesday, November 5th, to make plans for the meeting. A strong local committee was organized, of which Mr. Frederic Stanger was elected chairman. The committee has held several meetings since the above date and sub-committees on hotel reservations, reception and divisional meetings have been formed.

At this convention meetings will be held in sections for the first time. The Terra Cotta, Refractories, Glass and Enamel Divisions have already requested separate rooms for the reading and discussion of papers. It is planned to have a general meeting on Monday, February 23rd, and Divisional sessions on Tuesday, February 24th.

Members of the Society are urged to make hotel reservations at once for the week of the meeting.

Further details in reference to the meeting may be secured by writing to Mr. Frederic Stanger, Enterprise White Clay Company, Philadelphia, Pa.

Those wishing to contribute papers to the program of this meeting should communicate with Mr. R. H. Minton, Chairman, Committee on Papers and Programs, General Ceramics Co., Metuchen, N. J.

1920—Summer Meeting.

The following notice has been issued by the English Ceramic Society:

"The American Ceramic Society has sent a cordial invitation to our Society to visit the States in 1920. A special executive committee has been appointed by our American friends to arrange works' visits, etc. This invitation has given great pleasure to our

members and our Society has appointed Mr. Arthur Heath the Honorary Organizing Secretary on this side.

It is anticipated that the excursion will occupy a little less than a month. Arrangements will probably be made for joint discussions on subjects of special interest.

Please make all communications respecting the excursion to Mr. Arthur Heath, Central School of Science and Technology, Stoke-on-Trent."

It is planned to hold the Summer Meeting of the American Ceramic Society at the time of the visit of the English Ceramic Society, so that joint sessions of the two Societies may be held. A definite time for holding the meeting has not been decided upon, although it will probably be some time during the month of September.

The Glass Division of the American Ceramic Society has invited the English Society of Glass Technology to hold a joint meeting during the latter part of the summer of 1920. It is hoped that these dates may be made to coincide with those of the visit of the English Ceramic Society. The time tentatively set for the visit of the Society of Glass Technology is the early part of September, as during August glass plants are usually shut down because of the excessive heat at that time. An additional reason for postponing the meeting until September is that the Exposition of Chemical Industries will be held in New York during the week of September 20th. An opportunity to visit the Exposition will be an added inducement for our English friends to visit this country.

Definite plans are now being formulated for the itinerary and entertainment of our English friends during their visit to this side. These will be announced at an early date.

ERRATUM.

J. Am. Ceram. Soc., 2, 807, Oct. 1919. In Table 1, left hand column, the positions of Whiting and Feldspar should be reversed.

JOURNAL
OF THE
AMERICAN CERAMIC SOCIETY

A monthly journal devoted to the arts and sciences related to the silicate industries.

Vol. 2 **December, 1919** **No. 12**

EDITORIALS.

CHANGE OF EDITORSHIP.

The Committee on Publications regrets to announce the resignation of Editor George H. Brown which has been necessitated by the increasing pressure of his duties in connection with the Department of Ceramics at Rutgers College and the supervision of extension courses in the pottery center of Trenton. Mr. Brown's ability as Editor has been well demonstrated by the manner in which he has handled the publication of the Journal since its beginning in July, 1918, at a time when conditions required considerable initiative, resourcefulness and professional ability to launch a new technical publication.

On behalf of the American Ceramic Society the Committee on Publications desires to express appreciation of Mr. Brown's services in connection with the publication of Volumes 1 and 2 of this Journal, to wish him increasing success in the field of ceramic education in which his efforts are now to be concentrated and to hope that he will be a frequent contributor to the technical matter of the Journal, in the establishment of which his editorial services have been of the highest value.

In seeking a successor to Mr. Brown, the Board of Trustees has been fortunate in securing the services of Mr. Homer F. Staley of the Bureau of Standards, Washington, D. C. Mr. Staley is a graduate of Ohio State University, formerly director of the Department of Ceramics at Iowa State College, has had much experience in industrial lines, was editor of Volumes XIV and XV of the *Transactions of the American Ceramic Society* and is a past President of this Society. Mr. Staley is at present

Chief of the Sections of Enameled Metals and Refractories in the Bureau of Standards.

Mr. Staley is well equipped professionally and practically for his new duties as editor and the Committee on Publications looks forward to progressive success in the policies governing the publication of the Journal under his editorial direction which will commence with Volume 3, Number 1 or the January number of 1920.

L. E. BARRINGER,
Chairman, Committee on Publications.

DIVERSIFICATION OF TECHNICAL CONTRIBUTIONS TO THE JOURNAL.

In Volume 1, Number 12 of this Journal there was given a tabulation showing the diversification of the professional or technical papers published in the twelve numbers of Volume 1.

With the completion of Volume 2 by this number, a comparison can now be made between the diversification of papers in Volumes 1 and 2 and the following tabulation is submitted by the Committee on Publications for the information of our readers and contributors:

SUBJECT CLASSIFICATION.	Number of Papers. Vol. 1.	Vol. 2.
Clays, occurrence and uses	5	7
Refractories (fire-brick, saggers, retorts, glass-pots, etc.)	7	8
Porcelain and white ware	7	13
Glass	9	13
Lime and cement	5	4
Graphite crucibles	0	6
Enamels for metals	7	4
Enamels and glazes for clay wares	0	1
Terra cotta	4	0
Drying and driers	3	0
Firing and kilns	5	0
Miscellaneous (methods of testing and analysis, apparatus, construction, etc.)	..	..

From a study of the above table it is evident that to preserve a distribution of subjects consistent with the importance of the various phases of the ceramic industry, there should be more

contributions covering the fields of brick, tile and terra cotta and also regarding the technicalities of the important operations of drying and firing. This is a matter entirely in the hands of our members and readers and those desiring more matter in a given line should themselves either contribute such matter or solicit contributions from those they know are qualified to write upon such specific subjects.

The seeming preponderance of papers upon glass in Volume 2 was no doubt occasioned by the unusual activity during the war in investigating problems in the manufacture of optical glass. Upon the other hand—those interested in glass have been active from the outset in contributing professional papers to the Journal and it is to be hoped that they will retain their enthusiasm and their place.

The papers upon porcelain and white ware have been most largely upon porcelain and greater activity upon the part of those interested in white ware is highly desirable.

The appearance of a number of papers upon graphite crucibles in Volume 2 was the result of war-time activities in connection with the manufacture of refractories for metal melting. The technicalities of this field have been well-handled and a continuance of contributions of this class will be welcomed by our readers.

The field of ceramics is broad, its problems are interesting and everchanging, the men bearing the responsibilities of its progress are capable and professional pride and enthusiasm is well-developed. Under these conditions it is anticipated that the contributions to Volume 3 will be even more diversified and far-reaching than heretofore.

ORIGINAL PAPERS AND DISCUSSIONS.

THE MANUFACTURE OF ENAMEL LINED APPARATUS.

By Emerson P. Poste.

The field of vitreous enameling includes several rather distinct lines of manufacture. The broadest classification would no doubt have to do with the material to which enamel is applied and would divide the industry into steel enameling and cast iron enameling.

Under the general head of steel enameling the most familiar line is the manufacture of cooking ware[1] and similar commodities. Another branch of the industry which has assumed large proportions is the manufacture of signs, reflectors, table tops and other miscellaneous shapes. A third type of product, differing quite essentially from the other two in construction and use, is apparatus made of enameled steel designed to meet the needs of the dairy, canning, pharmaceutical and chemical industries.

The most prominent line of cast iron enameling is the manufacture of sanitary ware.[2,3] There are also a large number of miscellaneous lines involving the enameling of cast iron, among which may be named the manufacture of stove parts, barber and dentist chairs and various ornamental pieces. In addition to the above lines of cast iron manufacture there has been on the market for sometime a line of cast iron enameled apparatus which has found its particular application in the pharmaceutical and chemical

[1] Landrum, "Enamels for Sheet Steel," *J. Ind. Eng. Chem.*, **4**, 561 (1912).

[2] Staley, "The Manufacture of Enameled Iron Sanitary Ware," *Trans. Am. Ceram. Soc.*, **8**, 172 (1906).

[3] Staley, "Preparation and Application of Enamels for Cast Iron," *J. Am. Ceram. Soc.*, **1**, 534 (1918).

industries.[1] Equipment of this type was largely imported prior to the war but is now being put on the market by various American manufacturers.

It is our present purpose to deal with the manufacture of steel and cast-iron apparatus including some details relative to the other types of enamels, largely by way of contrast.

There are various ways in which the manufacture of enameled apparatus may be considered. It will be our choice to outline first the manufacture of the steel piece to which the enamel is

FIG. 1.—Plate in bending rolls.

to be applied, followed by a discussion of the manufacture of the cast iron piece. The question of enamel composition and preparation will be taken up, followed by the application and firing of the enamel on the steel or cast iron.

After considering some of the typical forms and uses of the

[1] "Acid Resisting Enamel Ware," *J. Am. Ceram. Soc.*, 1, 74 (1918).

finished apparatus a few points will be presented relative to rather technical features in connection with the manufacture and properties of enamels.

The Steel Shape.

The raw material which is to be formed into the final steel apparatus consists essentially of basic open-hearth plate, usually

FIG. 2.—Oxyacetylene welding.

a quarter of an inch in thickness, sometimes thinner, sometimes thicker, while in other lines of steel enameling the material may be thin sheets of steel varying from 24 to 16 gauge.

The apparatus consists generally of circular units, closed or

open top, jacketed or unjacketed, with various openings and fittings. The design of the piece of equipment to be made having

Fig. 3.—Grinding off weld.

been specified in the blue-print, sheets of the proper size are laid out in accordance with the drawing. The heads are then dished

to the specified depth and diameter and the plate, after trimming to the desired size, is put through the bending rolls and formed to the desired diameter.

The resulting cylinder is clamped in position and the longitudinal seam made by means of an oxyacetylene weld. Next,

FIG. 4.—Sandblasting a rectangular tank.

the bottom of the tank is attached to the shell in like manner and if it is to be a closed tank the top is welded on. The necessary openings in the form of inlets, outlets, manholes, etc., are then completed and the tank is ready to be prepared for enameling.

It is essential that the surface to be enameled be uniform

and clean and the first step to this end involves the grinding down of the weld by means of portable electric grinders.

The next step is the sandblasting of the entire surface of the unit so that wherever the enamel is to be applied there may be a surface free from impurities.

The sandblasting operation consists essentially of forcing a stream of sharp quartz sand against the surface to be cleaned. This is accomplished by passing air under pressure through a proper mixer at the bottom of a hopper in which the sand is stored. The air carries the sand through to the nozzle at a high rate of speed and causes it to be very forcibly thrown against the surface being treated.

In the case of cooking ware, or other thin steel products, the blanks are shaped either by pressing, spinning, or drawing, proper dies for such operations having previously been made. In preparation for enameling, the ware is thoroughly pickled and in some cases annealed in an annealing furnace. This operation accomplishes the removal of a considerable amount of scale which greatly facilitates the pickling which is to follow.

The Cast Iron Shape.

The blue-print, showing all the details necessary to the design of the piece of cast iron equipment to be enameled, goes to the pattern shop at which point the necessary patterns are produced, these in turn going to the foundry where the pieces are cast. The quality of the casting must be very good in order that a proper surface may be presented for receiving the enamel.

After leaving the foundry a certain amount of machine work is necessary to bring portions of the casting to proper dimensions, following which the piece goes to the sandblast and receives a treatment practically the same as that received by the steel apparatus.

The Enamel.

The preparation of the enamel which is to be applied to the ware, whether steel or cast iron, involves essentially three steps; mixing, smelting, and grinding.[1]

As to the formulas from which various enamels are prepared,

[1] *Loc. cit.*, *J. Am. Ceram. Soc.*, 1, 534 (1918).

it may be of interest to note certain typical compositions covering a rather large range of enameling technology.[1,2,3]

We herewith give the following formulas (Tables 1 and 2): No. 1 a steel ground coat; No. 2 a steel cover coat, white; No. 3 a cast iron ground coat; No. 4 a cast iron cover coat, white, all expressed in terms of the raw materials,[4] used for mixing.[5,6,7,8,9,10,11,12,13]

In the following tables the term "ground coat,"[5,6,7,8,9] in either case refers to the particular type of enamel which is applied to the freshly prepared metal while the term "cover coat,"[2,5] refers to the enamel which is applied above the ground coat and gives the finished appearance to the ware.

The mixing of the raw batch in accordance with a given formula may be accomplished by one of several means including shoveling, similar to the method used for mixing small amounts of concrete, or some type of mechanical mixing in which a whole batch is given

[1] Landrum, "A Comparison of Ten White Enamels for Sheet Steel," *Trans. Am. Ceram. Soc.*, **14**, 489 (1912).

[2] Riddle, "Types of Enamel Used for Enameling Cast Iron Sanitary Ware," *Ibid.*, **9**, 646 (1907).

[3] Staley, "Enamels for Cast Iron," *J. Am. Ceram. Soc.*, **1**, 703 (1918).

[4] Landrum, "The Function of the Various Raw Materials in a Sheet Steel Enamel," *Eighth International Congress of Applied Chemistry*, **25**, 317.

[5] Shaw, "The Allowable Limit of Variation in the Ingredients of Enamels for Sheet Steel," *Trans. Am. Ceram. Soc.*, **11**, 103 (1909).

[6] Landrum, "The Necessity of Cobalt Oxide in Ground-Coat Enamels for Sheet Steel," *Ibid.*, **14**, 756 (1912).

[7] Danielson, "The Effect of Variation in the Composition of Ground Coats for Sheet Iron Enamels," *Ibid.*, **18**, 896 (1916).

[8] Coe, "A Comparison of Commercial Ground Coats for Cast Iron Enamels," *Ibid.*, **13**, 531 (1911).

[9] Staley, "Ground Coat Enamels for Cast Iron," *J. Am. Ceram. Soc.*, **1**, 99 (1918).

[10] R. E. Brown, "Replacement of Tin Oxide by Antimony Oxide in Enamels for Cast Iron," *Trans. Am. Ceram. Soc.*, **14**, 740 (1912).

[11] Shaw, "Antimony Oxide as an Opacifier in Cast Iron Enamels," *J. Am. Ceram. Soc.*, **1**, 502 (1918).

[12] Staley-Fisher, "Leadless Enamels," *Trans. Am. Ceram. Soc.*, **15**, 620 (1913).

[13] Staley, "Antimony Compounds as Opacifiers in Enamels," *Ibid.*, **17**, 173 (1915).

a thorough treatment, somewhat similar to the mixing of large batches of concrete.

TABLE I.

Batch Formulas.

Raw materials.	No. 1. Steel ground coat.[a]	No. 2. Steel cover, white.[b]	No. 3. Cast iron ground coat.	No. 4. Cast iron cover, white.[c]
Soda	74	...	...	71
Borax	356	154	288	133
Saltpeter	...	65	16	...
Chili saltpeter	...	...	...	24
Lead oxide	...	...	...	144
Zinc oxide	...	...	...	44
Tin oxide	...	...	...	73
Calcspar	...	65	...	24
Barium carbonate	...	...	...	11
Magnesium carbonate	...	10	...	...
Feldspar	363	386	...	372
Fluorspar	53	13	...	107
Cryolite	...	117	...	...
Quartz	144	190	700	...
Manganese dioxide	$6^1/_2$	...	...	...
Cobalt oxide	$2^1/_4$	...	...	...
Mill additions	6% clay 2.5% borax	12% Tin oxide 7% clay $^1/_4$% Mg oxide	9% Sand 12% clay	None

[a] *Trans. Am. Ceram. Soc.*, **14**, 759 (1912).
[b] *Ibid.*, **14**, 498 (1912).
[c] *Ibid.*, **15**, 622 (1913).

The raw batch is introduced into what is termed a "smelting furnace" which is essentially a reverberatory furnace. Reactions take place on the hearth of the smelter involving the various thermal decompositions and combinations characteristic of the particular raw materials involved.[1] After the enamel has been reduced to a uniform fusion the furnace is tapped and the charge run into a trough of cold water, resulting in a thorough shattering

[1] Poste-Rice, "Effect of the Degree of Smelting upon the Properties of a Frit," *J. Am. Ceram. Soc.*, **1**, 221 (1918).

of the product giving a material known as "frit." It is customary to assume in the composition of this frit that the amounts of the various non-volatile oxides involved in the original batch have entered into a combination which is expressed in terms of the molecular weights of the component parts by what is known as the "molecular formula." In Table 2 will be found the molecular formulas of batches given in the first part.

TABLE 2.

Molecular Formulas.

	No. 1. Steel ground coat.	No. 2. Steel cover, white.	No. 3. Cast iron ground coat.	No. 4. Cast iron cover, white.
Na_2O	0.64	0.50	.....	0.30
K_2O	0.08	0.18	.....	0.09
CaO	0.24	0.28	.....	0.35
BaO	..	..	.....	0.01
MgO	..	0.04	.....	..
PbO	..	..	.....	0.13
ZnO	..	..	.....	0.12
MnO	0.03	..	.....	..
CoO	0.01	..	.....	..
Al_2O_3	0.20	0.30	.....	0.15
SiO_2	2.26	2.51	.....	0.86
B_2O_3	0.63	0.26	.....	0.15
F_2	0.23	0.60	.....	0.30
SnO_2	..	..	.....	0.10

As a matter of fact it has been found that, while the actual composition of the frit approaches the theoretical composition as expressed by the molecular formula, there are actually variations depending largely upon the degree of smelting. The more volatile materials such as soda, potash and boric oxide are driven off to a greater or less extent and the result is that the composition of the final frit is lower in these materials than theoretically called for.[1]

There is an exception to the above general scheme of smelting which involves the processing of one type of cast iron ground coat. In this particular case the raw materials are thoroughly mixed but instead of being fused to a liquid state on a smelter

[1] *Loc. cit.*, *J. Am. Ceram. Soc.*, **1**, 221 (1918).

hearth they are placed in a cast iron tray and heated to a bright red and thoroughly stirred at intervals. The result is a sintered mass closely resembling pumice stone. This must be further reduced by crushing before being ground for application to the ware.

FIG. 5.—Spraying a steel tank.

The next step in the processing of the enamel is the grinding, which may be accomplished either wet or dry. While there are variations to the general statement, we will assume that the wet grinding is for steel enamels and cast iron ground coats only, and dry grinding for cast iron cover coats.

In the wet grinding of the enamel the object is to secure a suspension of the enamel in a finely ground condition so that it may be treated essentially as a liquid. This is accomplished by grinding in a pebble mill a certain amount of frit in the presence of a fixed amount of water and various other mill additions. A necessary material is clay, which has the property of holding the enamel particles in suspension and is susceptible to further

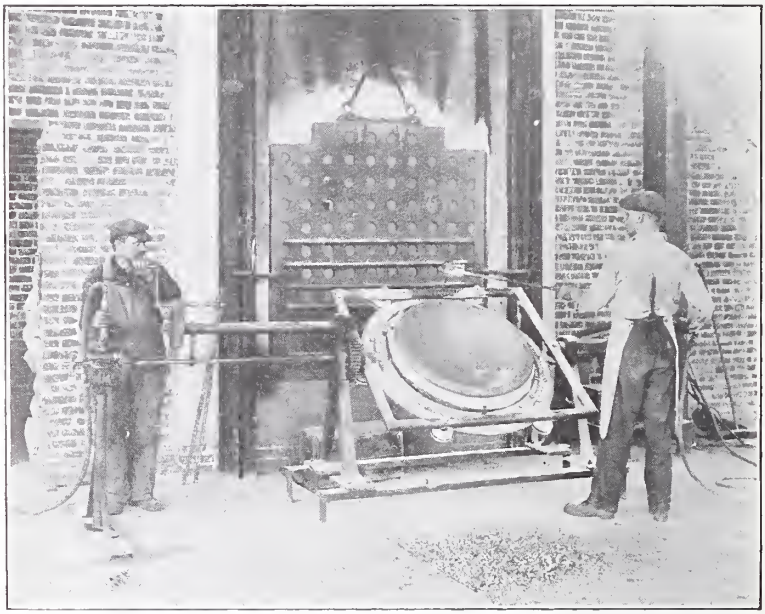


FIG. 6.—Dredging a cast iron dish.

treatment termed "setting up" rendering the suspension particularly stable.[1,2] In the grinding of steel ground coats and some other types of enamel, clay forms the only mill addition, but to produce various colors, materials are often added in the mill which accomplish the desired results. For instance, in the manufacture of white enamel for cooking ware, the customary mill addition is tin oxide.

[1] *Loc. cit., Eighth International Congress Applied Chemistry,* **25,** 317.

[2] Shaw, "American Clays for Floating Enamels," *Trans. Am. Ceram. Soc.,* **19,** 339 (1917).

In some cases it is desirable to control the melting point of the enamel on the ware by adding refractory materials, such as silica or feldspar, in the mill. It is further a frequent practice to introduce into the mill a small amount of material to accomplish the "setting up" of the enamel, further reference to which will be made later. Magnesium oxide and borax are substances frequently used at this stage. In the above table of formulas the various mill additions are given.

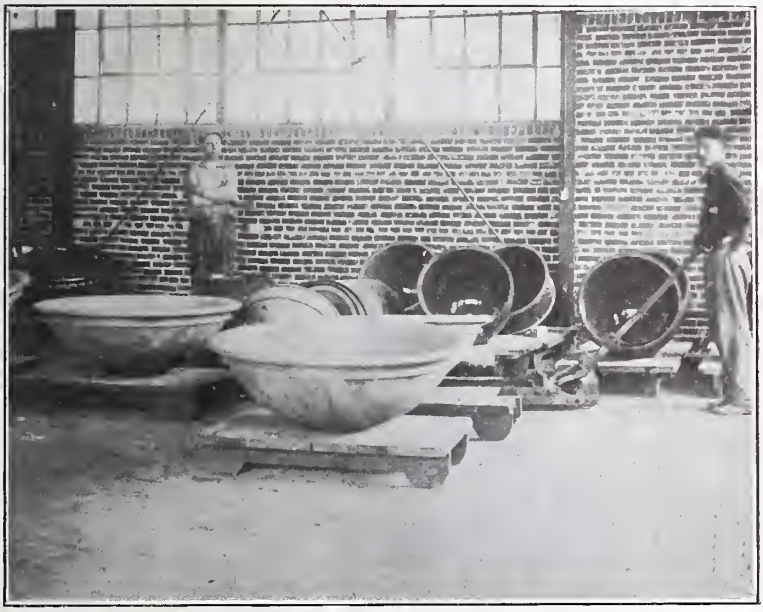


FIG. 7.—Cast iron pieces in process.

After the enamel has been properly ground it is drawn from the mill and stored in containers ready for delivery to the enameling department.

Application.

Steel.—A further treatment will be necessary before the enamel is ready for application to the ware. If the enamel, as it comes from the mill, is diluted to the proper consistency for application,

the suspending power of clay will not be sufficient to prevent settling but if an electrolyte be added in proper amounts a very definite change in the physical properties of the suspension will take place, making it possible to vary the consistency of the enamel through quite a large range without causing settling. A material added for this purpose is termed a "vehicle" and several compounds are used. Rather common practice involves the use of borax for "setting up" the ground coat and magnesium sulphate for the cover coat.[1]

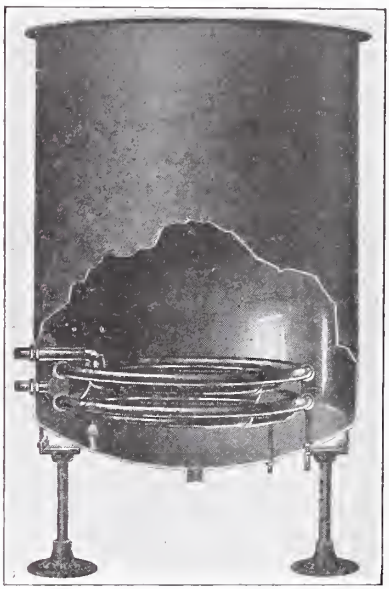


FIG. 8.—Open-top tank with heating coil.

The enamel may be applied to the ware in one of several ways. If the shapes are small and are to be enameled on both sides they may be "dipped" into a relatively large tub of the prepared enamel, the excess enamel being shaken off and the piece rapidly dried. If the enamel is to be applied to but one side it may be poured over the surface which is held at a proper angle so that the excess drains off. This is termed "slushing." In cases where

[1] *Loc. cit., Trans. Am. Ceram. Soc.*, **19**, 339 (1917).

dipping or slushing would be impractical "spraying," which consists essentially of spraying the enamel by means of a compressed air atomizer, is resorted to. This method of application is the one used in the manufacture of steel apparatus of large dimensions.

Burning.

Steel.—After the ground coat has been applied and dried the ware is ready for burning which is accomplished in a furnace, the temperature of which ranges from 1600° to 2200° F., depending

FIG. 9.—Plain closed-top tank, with off-center agitator.

upon the particular type of ware and enamel being burned. A given ground coat requires a higher temperature than the cover coat which is applied to it, and pieces made of heavy metal call for higher temperatures than pieces made of light metal.

Furnaces may be classified as "muffle" furnaces or "direct fire" furnaces, the essential difference being that in the former case the ware is placed in an inner chamber which is surrounded

by the products of combustion while in the latter the products of combustion actually come in contact with the ware being burned. The first type of furnace is generally used in the manufacture of cooking ware and other small pieces while the direct-fire furnace is commonly employed in the manufacture of larger apparatus.

FIG. 10.—Jacketed open-top tank.

When a piece of ware is placed in the furnace certain preliminary reactions take place, such as the dehydration of the clay and any other mill additions which in drying may have retained moisture. The individual particles of the enamel then melt and run together forming the finished surface. After the ground coat has been burned and allowed to cool another cover coat is

applied in a similar manner, dried and burned and the process repeated until the desired amount of enamel has been applied.

FIG. 11.—Jacketed closed-top tank with high speed side agitator.

In the case of steel apparatus the piece is now ready for assembling which may involve the attachment of the supports or per-

haps the fastening on of the steam jacket and equipping with agitators, thermometers, pressure gauges, relief valves and other accessories.

Cast Iron.—In the enameling of the cast iron shapes a ground coat is applied, generally by slushing for sanitary ware and by spraying for apparatus. This coat is thoroughly dried and the ware is placed in a muffle furnace and brought up to a uniform temperature approaching a bright red. In the case of the sintered

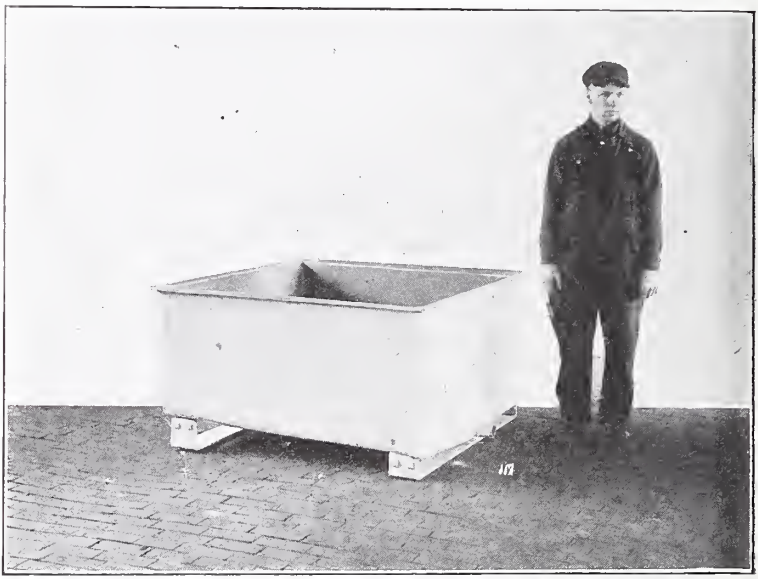


FIG. 12.—Rectangular tank.

ground coat the ware at this point if allowed to cool would present a rough surface while in the case of a fritted ground coat the enamel would be glossy.

However, the piece is not permitted to cool when withdrawn from the furnace. The enamel to be applied has been ground to a very fine dry powder by placing the dry frit in a mill and grinding without mill additions. The proper amount of this fine powder is placed on a sieve which, together with a handle and vibrator operated either by compressed air or electricity, is

termed a "dredge." By the use of this piece of apparatus the fine powder is shaken on the hot surface of the ware, which, after being withdrawn from the furnace, has been placed on a "turn table." This machine is so constructed that ware placed on it can be rotated and tipped up and down to any position. After a proper amount of the enamel has been applied to the revolving piece it is run back into the furnace and allowed to come to a sufficient temperature to fuse the enamel down to a fairly uniform coat. The piece is drawn out and another layer of enamel applied, put back into the furnace and burned down and the

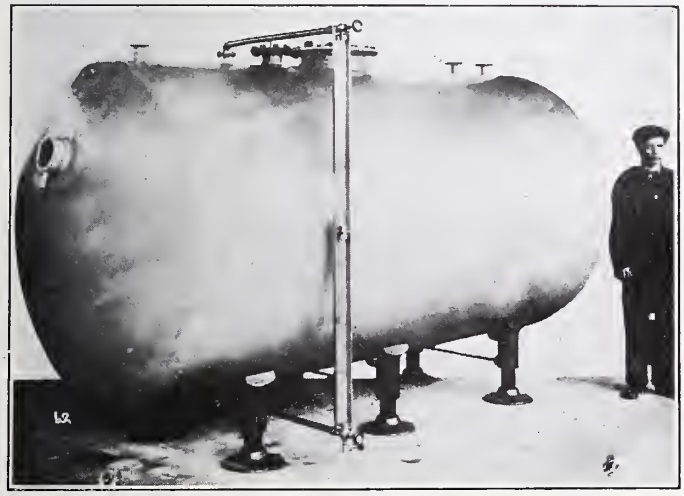


FIG. 13.—Horizontal storage tank.

operation repeated until the desired thickness is produced, after which it is given a firing somewhat harder than the previous ones, the result being a very smooth surface with a high gloss.

The piece of ware thus processed is ready for assembling, which may involve any or all of the operations referred to in connection with the assembling of the steel apparatus, depending upon the form of equipment being made, which may range from an unjacketed, open-top dish to a jacketed, vacuum pan with agitator and other accessories.

Uses of Enameled Wares.

The uses to which enamel lined apparatus are put cover a large field ranging from the mild conditions characteristic of the dairy industries to such severe chemical conditions as dilute mineral acid. In general the steel apparatus, available in much larger units than the cast iron, finds its application under the milder conditions while the cast iron apparatus, enameled with an entirely different formula, proves of service under the more extreme conditions.

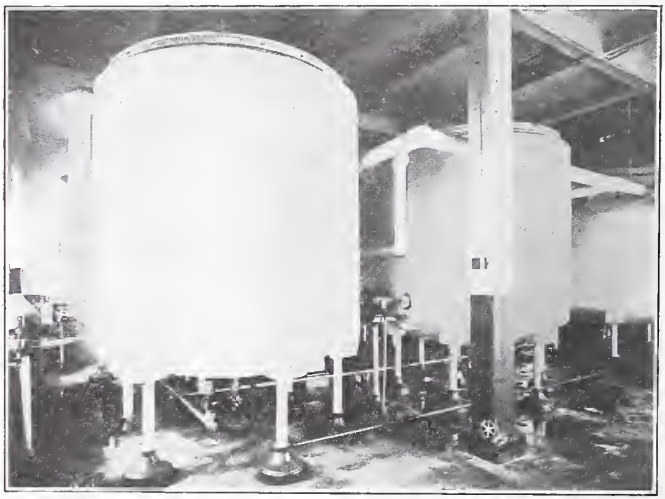


FIG. 14.—Six 2500 gallon tanks for milk storage.

The actual construction of the steel apparatus may include tanks up to ten feet in diameter and about twelve feet in length, which may be either open or closed top, jacketed or unjacketed, with or without agitator, the design varying to meet the particular conditions involved.

In dairy operations there are a great many uses for this line of material, such as holding and cooling of milk and aging of ice cream, while in the canning and other food preparation industries there are various forms of cooking kettles, vacuum pans and

storage units. In pharmaceutical work this line of equipment is very efficient for the mixing and storage of various preparations.

The cast iron equipment, available in the form of shallow dishes and pans, open-top kettles, jacketed or unjacketed, stills, vacuum pans and digestors, is used primarily in connection with the pharmaceutical and chemical industries where conditions are such as to render it impossible to carry on the desired processes in contact with metal.

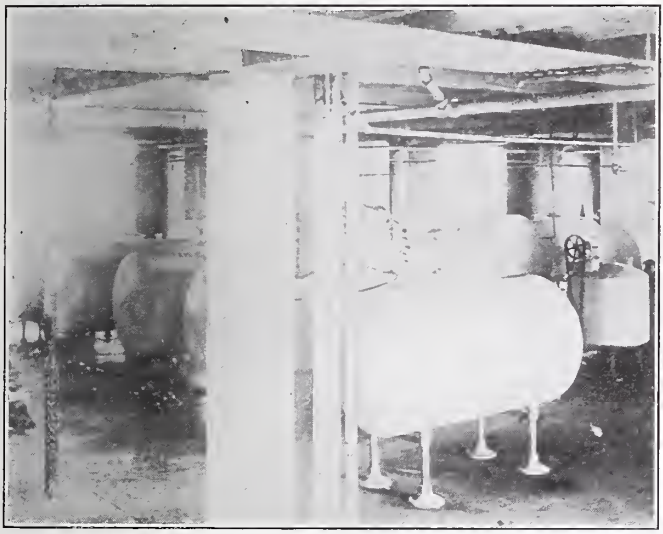


FIG. 15.—Storage of pharmaceutical preparations.

Technical Control.

It would be quite impossible to enumerate all of the features of the technical control of an enameling operation. Of prime importance in all cases is the quality of the raw materials which are being used. The steel should be a basic open hearth steel of low carbon content and in such mechanical condition that it will not develop cracks or other imperfections which will cause corresponding defects in the enamel. The cast iron should be a good quality of gray iron free from mechanical defects.

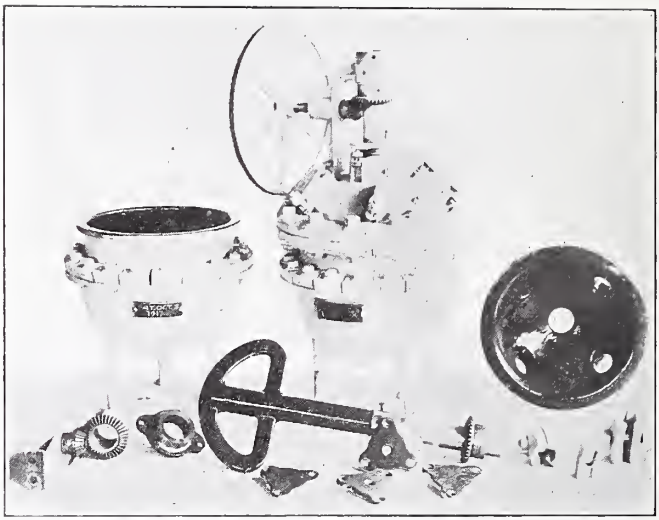


FIG. 16.—Cast iron laboratory autoclave.

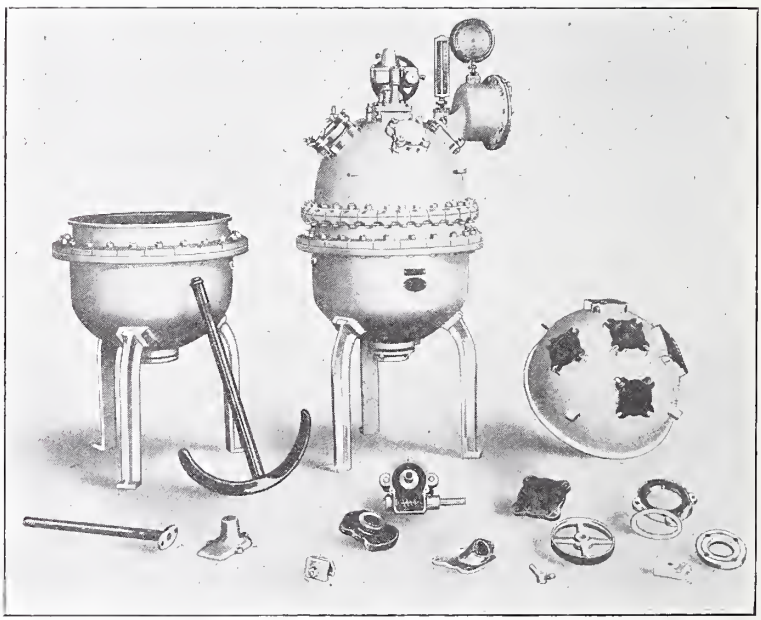


FIG. 17.—Cast iron still with agitator and accessories.

Uniformity of raw materials for the enamel or correction of formulas in the case of irregularities are fundamental if the product is to be uniform. This necessitates an analysis of the raw materials.[1,2] A failure to appreciate the possible variations of raw materials and the consequent results are a very serious matters. As an extreme case we know of a particular instance in which an enameler was using borax which had been stored in bags quite close to a furnace. The theoretical composition of borax and the composition which will be quite closely approached

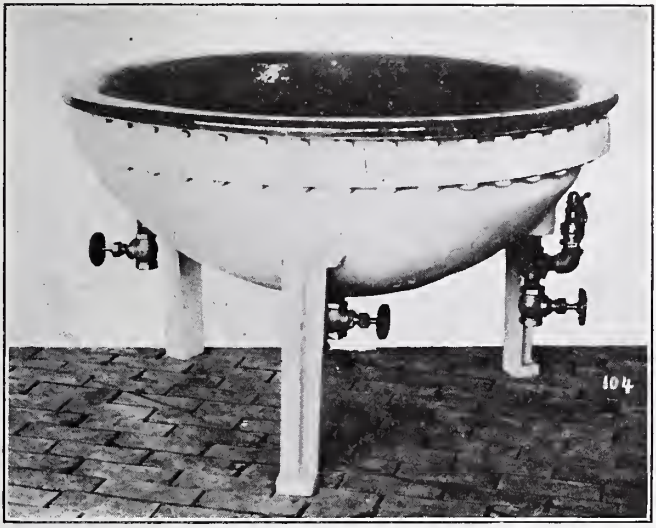


FIG. 18.—42″ jacketed cast iron evaporating dish.

under normal storage conditions is approximately 53 per cent sodium tetraborate, the balance being water of crystallization. In the case noted the sodium tetraborate figure ran very close to 70 per cent. In other words, for every 100 lbs. of borax taken, 70 lbs. of sodium tetraborate were introduced rather than 53 lbs. At this particular time the enamel was running very soft and overburned very readily. The explanation is obvious.

[1] Landrum, "Methods of Analysis for Enamels and Raw Materials," *Trans. Am. Ceram. Soc.*, 12, 144 (1910).

[2] J. W. Mellor, "Treatise on the Ceramic Industries," Vol. 1.

The materials which are particularly subject to variations in composition and which, at the same time, enter into all enamel

FIG. 19.—Cast iron open-top boiling kettle.

batches in considerable quantities are soda, borax, and feldspar. As has been pointed out, the borax is susceptible to the loss of

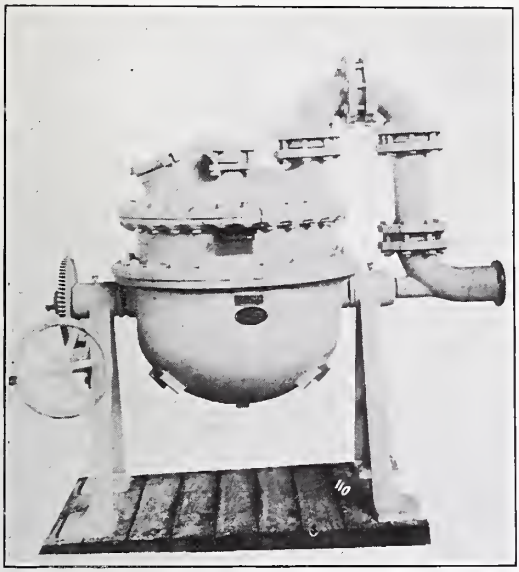


FIG. 20.—Cast iron jacketed closed-top tilting kettle with fume vent.

FIG. 21.—Group of various cast iron apparatus.

moisture and it has been found experimentally that when stored under very dry and warm conditions the water of hydration

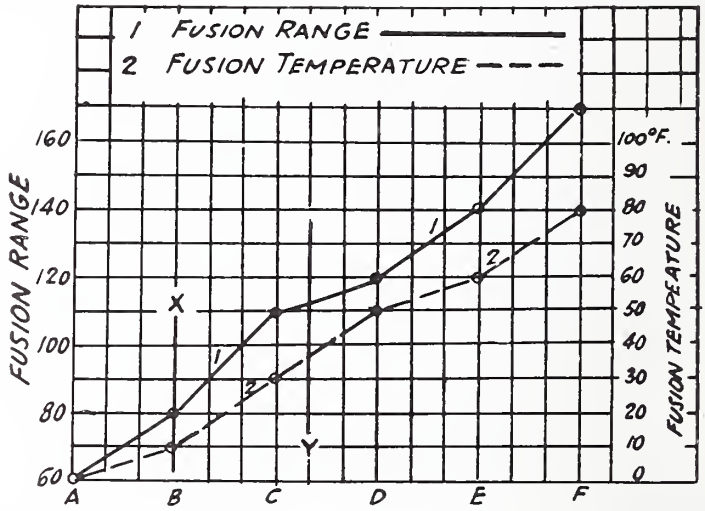


FIG. 22.—Increase of deformation range and rise of deformation temperature produced by continued smelting.

which should be present to the equivalent of 10 molecules of water can be reduced to 5 molecules of water and this lower

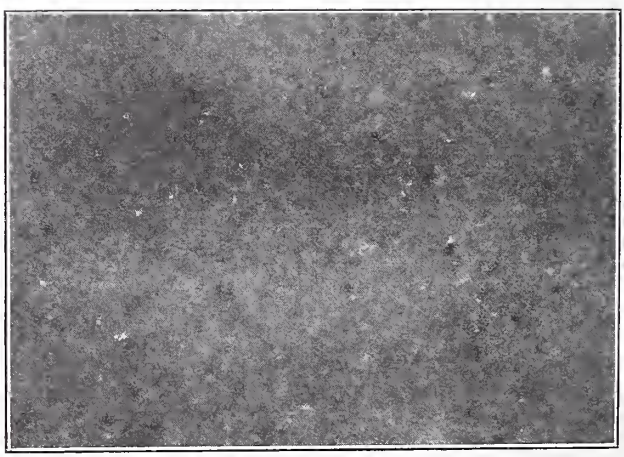


FIG. 23.—Underburned enamel. (75 diameters.)

hydrate, exposed to the atmosphere, will again take up the 5 molecules of water approaching the theoretical borax composition.

Sodium carbonate will absorb moisture and carbon dioxide from the air so that in some cases a greater weight than that theoretically called for must be taken to introduce a given amount of soda.

Feldspar may vary quite materially in the alkalies and again will contain as an impurity various amounts of free silica, a difference which may make quite radical variations in the enamel. Based on the same specified quality of feldspar we have received

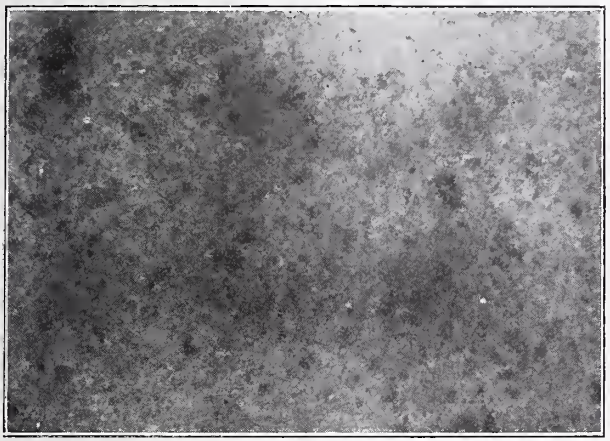


FIG. 24.—Normal burned enamel. (75 diameters.)

material ranging from 65 to 75 per cent silica, an irregularity which, if neglected, will cause rather radical differences in the final product.

The matter of smelting, grinding and burning are details which must be controlled somewhat differently under one set of conditions than under another. In other words, those in charge must become familiar with the particular type of enamel, determining what particular degree of smelting, fineness of grinding and burning temperatures produce the best results. The use of

definite fineness tests and accurate temperature control are quite essential if uniformity is to be realized.

The properties of the final enamel, both physical and chemical, are points which have to do with its service in the field and are properties which are very often tested.

There are various physical properties both of the frit and the final enamel which are worthy of attention and which may vary through wide ranges with a given formula if not properly processed. Under- or oversmelting will produce radical differences as to the fusion[1] temperature of a given formula and over- or

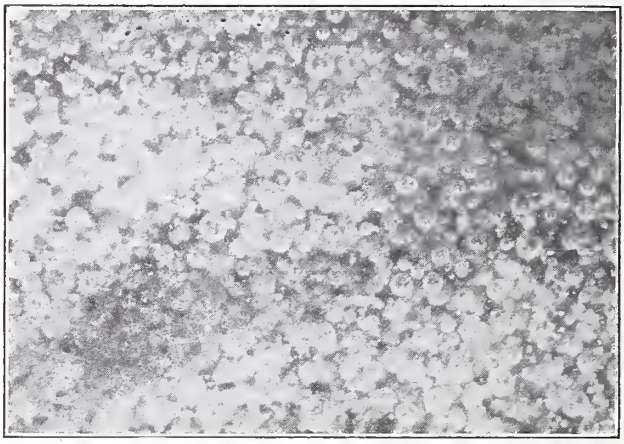


FIG. 25.—Overburned enamel. (75 diameters.)

underburning will produce irregularities both as to the actual nature of the surface exposed and the ability of the enamel to adhere to the metal.

The underburned enamel when examined under the microscope will show a rather irregular condition involving the presence of unfused particles while the overburned condition will show a very serious tendency toward the formation of large bubbles. The intermediate stage approximating the proper burn will show practically no unfused material and the first appearance of fine bubbles.[2]

[1] *Loc. cit., J. Am. Ceram. Soc.*, 1, 221 (1918).

[2] Poste, "Enamel Surfaces under the Microscope," *Trans. Am. Ceram. Soc.*, 19, 146 (1917).

The ability of the enamel to adhere to the metal under mechanical abuse has been the subject of various methods of testing, none of which can be taken as entirely satisfactory.[1] There are three different conditions which should be considered in a me-

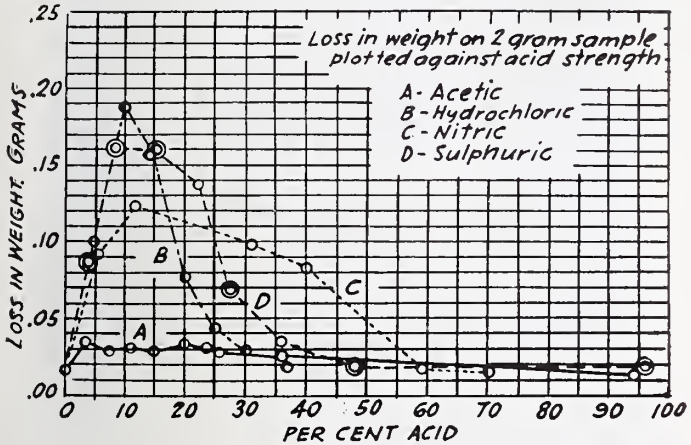


Fig. 26.—Relative activity of acids on enamel.

chanical test of the impact type. The blow on the enameled surface may not be sufficient to deflect the steel in which case it amounts to a crushing or pulverizing test of the enamel itself. The blow may be sufficiently severe to cause a momentary de-

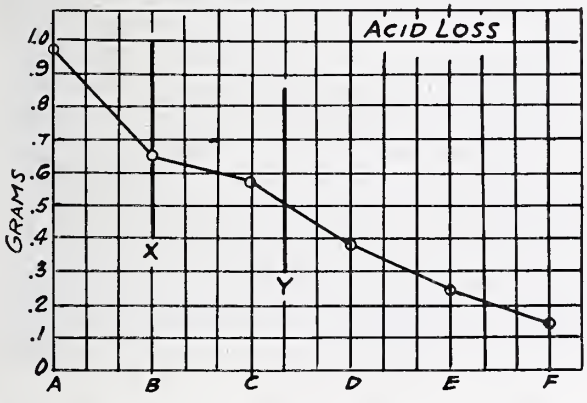


FIG. 27.—Decrease of activity of acid on enamel with continued smelting.

[1] *Loc. cit.*, *Trans. Am. Ceram. Soc.*, 14, 489 (1912).

flection of the steel within its elastic limit following which the steel will resume its normal position without setting up permanent

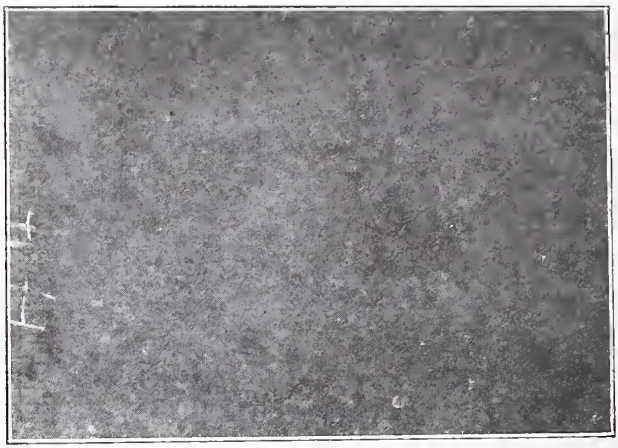


FIG. 28.—Underburned enamel after etching with acid (75 diameters.)

strains in the metal although there may be a failure of the enamel. Or the steel may be deflected beyond the elastic limit giving a

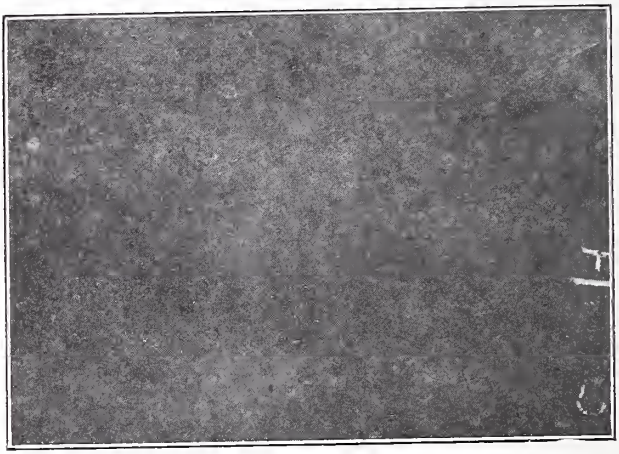


FIG. 29.—Normal burned enamel after etching with acid. (75 diameters.)

permanent deformation in which case the relation of the enamel to the steel is materially different from under either of the other conditions.

A chemical property of the enamel which is a matter of prime importance is its ability to withstand the corrosive or solvent action of the substances with which it is to come in contact, and especially in connection with apparatus to be used in the chemical industries. There are various means of determining and comparing the resistance of enamels to such action some of them having to do with the frit[1,2,3] and others with the final ware.[4]

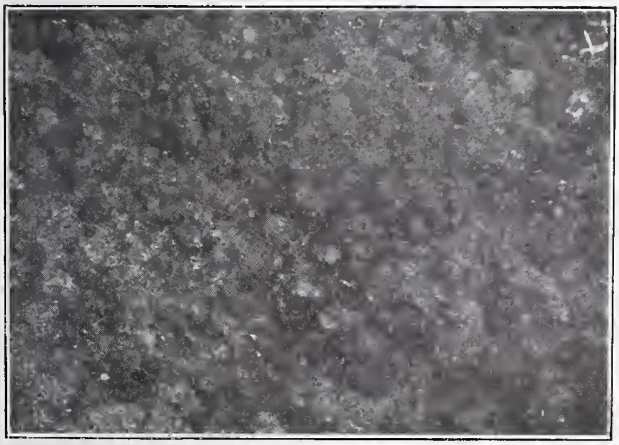


FIG. 30.—Overburned enamel after etching with acid. (75 diameters.)

It is quite evident from the work that has been done that no satisfactory method exists which will enable various people under different conditions to obtain comparative results and the Committee of Standards of the American Ceramic Society has

[1] Poste, "The Relative Action of the Acids on Enamel," *Trans. Am. Ceram. Soc.*, **17**, 137 (1915); "The Relative Action of the Acids on Enamel," *Ibid.*, **18**, 762–926 (1916); "The Relative Action of the Acids on Enamel," *J. Am. Ceram. Soc.*, **2**, 32 (1919).

[2] Frost, "Action of Acetic Acid Solutions of Different Strengths on Sheet Steel Enamel," *J. Am. Ceram. Soc.*, **1**, 422 (1918).

[3] Landrum, "The Resistance of Sheet Steel Enamel to Solution by Acetic Acids of Various Strengths," *Trans. Am. Ceram. Soc.*, **13**, 494 (1911).

[4] *Loc. cit.*, *Trans. Am. Ceram. Soc.*, **14**, 489 (1912).

appointed a Sub-Committee on Enamels which is working on this problem at the present time.

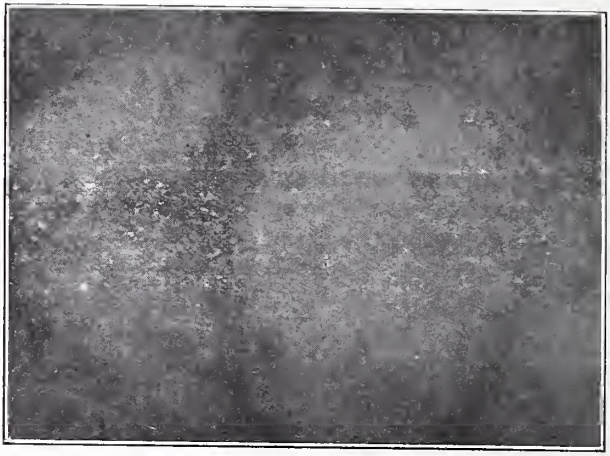


FIG. 31.—Mottled cooking ware. (75 diameters.)

In a general way it may be stated that the action of a given acid on an enamel is the stronger the greater the ion concentration

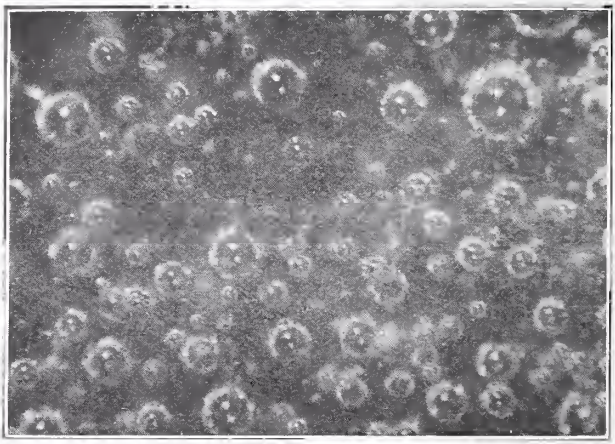


FIG. 32.—Overburned commercial ware. (75 diameters.)

or electrical conductivity of the acid.[1] For example, it is a well-established fact that dilute hydrochloric acid is far more

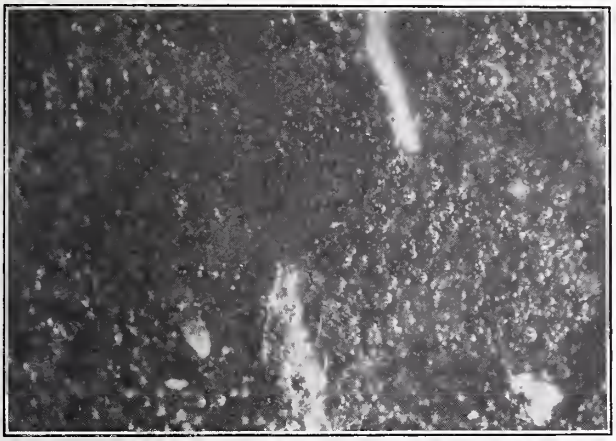


FIG. 33.—Commercial ware. (75 diameters.)

active than concentrated acid. It is very evident that details in processing may materially vary the acid resistance of a given enamel in its final form. In the first place the harder the smelting

Fig. 34.—Very homogeneous commercial ware. (75 diameters.)

[1] *Loc. cit.*

the greater the acid resistance because of a change in the chemical composition of the frit itself. The manner in which the enamel is burned on the ware also has a bearing on its acid resistance. This point has been definitely proven by observing through the microscope enamels which have been under- and overburned and then etched with acid, the overburned enamel having been subject to much more decomposition than the underburned, probably as a result of the different physical conditions existing at the surface due to the formation of large bubbles previously mentioned.[1]

The question of under- and overburning as affecting the final conditions of the enamel suggests noting the result of the examination of several pieces of commercial ware under the microscope, all magnified to the same diameter. Fig. 31 is a microphotograph of a mottled cooking ware enamel; Fig. 32 a commercial enamel which was apparently seriously overburned; Fig. 33 another piece of commercial ware; and Fig. 34 an exceptionally uniform commercial ware.

ELYRIA ENAMELED PRODUCTS CO.,
ELYRIA, OHIO.

[1] *Loc. cit.*, *Trans. Am. Ceram. Soc.*, **19**, 146 (1917).

COMPARISON TESTS FOR STRIAE IN OPTICAL GLASS, BY THE BRASHEAR CONVERGING LIGHT, DIRECT VIEW METHOD, THE BUREAU OF STANDARDS TANK IMMERSION METHOD, AND THE SHORT RANGE PROJECTION METHOD.[1]

By L. E. Dodd.

I. Introduction.

A study of the manner in which light transmitted through optical glass is able to make visible the striae so often present in it as a very important defect, leads to the conclusion that the visibility of the striae is highest where the light, assumed to be of suitable intensity, issues from the most concentrated source (so-called "point source") and is moreover monochromatic. Of the two requirements, concentrated source and monochromatism, the former would appear to be in general the more necessary until the source has reached the last stage of practicable concentration. In this last statement the question is omitted of obtaining a "match" between the liquid and the glass in an immersion method.

It has been common practice to employ a line source for purposes of striae detection, and this is no doubt practicable, if not so precise, where the glass sample under test can be freely turned so that it may be observed with the light passing through it in any desired direction. But the line source, as contrasted with a point source, is concentrated only relative to its own transverse direction, for in its own plane containing the eye of the observer—supposing the line source to be perpendicular to the line of sight—it is an extended source and acts as such. The result is non-uniform visibility of the striae in the sample, for only those components parallel to the line source are revealed distinctly, while the remainder, for the same position of the sample, are partially or wholly obscured.

[1] By permission of the Director, Bureau of Standards.

Although a turning of the piece of glass during inspection makes the line source method practicable to a certain degree, an absolutely free turning to permit the light to pass through the sample in many successively different directions is not so practicable for routine testing on account of the time required. The conditions of uniform visibility afforded by a point source are therefore desirable, so that for a given position of the sample the maximum number of parts of striae present can be seen. Although even with a point source some turning of the sample may still be necessary because of the lack of symmetry of the striae as to both their shape and orientation relative to the source, the amount of such turning should be considerably lessened where one of the conditions requiring it has been removed.

Given, then, a truly concentrated source of suitable intensity and in addition monochromatic light, the illumination is ideal for striae testing. The various possible methods of such testing belong to one or the other of two general types, what may be termed the direct view type, and the projection type. In the former the glass sample is viewed directly either with the unaided eye or with telescope or microscope, and in the latter type the light, which may be parallel, convergent, or divergent as it passes through the sample, is intercepted, after transmission through the glass, at either an opaque or a translucent screen, where a shadow pattern of the striae is formed and there viewed by the observer. Photography as it has been applied to exhibit striae in glass is to be included probably in all cases in the projection class.

Object of the Present Work.

In view of the more concentrated filaments in electric lamps of recent manufacture it has appeared that the projection method might readily be applied, at least for polished samples, to short ranges in a compact apparatus having neither prisms nor lenses. Such an apparatus would consist of three parts, light source, stand or other support for the glass sample, and projection screen. It seemed likely that its total length could be made only a foot or less. It was proposed to use a translucent screen. By this device the striae shadow patterns, or images, could be viewed by the observer from that side of the screen away from the sample

and the light source, thus reducing the space necessary for the apparatus while still keeping the position of the sample during inspection within easy reach of the observer. It was also proposed to test this method with white light illumination.

The primary object of the present work therefore has been to learn something definite of the possibilities of the short range projection method for detecting striae in optical glass. It was thought that one of the best ways to accomplish this would be by means of a comparison of striae methods in a test series carried out so that some thoroughness could be attributed to the work, sufficient at any rate to make the conclusions reliable.

Plan of the Work.

In order to make with more thoroughness a test of the general method of short-range projection, a number of series of comparison tests, five in all, were performed. For this purpose there was provided a number of glass samples regarded as large enough for a fair test. Thus 117 samples comprised the test set. The same set was used in each of the five series. These five comparison tests, each giving its own data, required four different set-ups of apparatus, employing, respectively, the Brashear[1] converging light, direct view method, the modified tank immersion method of the Bureau of Standards, and the short range projection method. The Bureau of Standards tank immersion apparatus was used with the samples both in the polished and the rough-ground states. The tank alone of this apparatus was used also with the short range projection method where the samples had ground surfaces.

The experimental arrangements are described later in this paper. By permission of Mr. J. B. McDowell the Brashear apparatus was employed for the first series, since it was readily available for comparison purposes and would serve to check the S. R. P. method and the T. I. method, as the latter has been regularly operated by the Bureau. It was used at the Brashear plant and was apparatus that had been employed there for some time. At that plant there is used also for precision test for

[1] By the "Brashear" method is here meant the direct view, convergent light method for striae testing as used with telescope objective by Brashear.

striae of large blocks of glass a spherical mirror method, into the merits of which the present work has not gone. The tank immersion apparatus of these tests was the one which has been in regular use by the government during the past seven or eight months, for routine testing in the optical glass factory at the Pittsburgh Branch of the Bureau of Standards. Apparatus for the S. R. P. method was set up at that plant.

Apparatus

The accompanying diagrams (Fig. 1) with legends may suffice for description of the experimental arrangements and illumination systems. Information with reference to the five comparative test series is tabulated below. A somewhat detailed verbal description of the light source and the translucent screen of the S. R. P. apparatus in the present work, and of the manner in which it is used, will be included under this head.

It is to be noted that tank immersion is merely an adjunct to either of the two general types of striae testing apparatus, being necessary where the glass surfaces are unpolished, to eliminate surface markings by making them invisible. This is accomplished by obtaining a "match" of indices of glass and liquid for the particular wave length of the monochromatic light used. Where the light is not monochromatic a perfect match cannot, of course, be obtained.

Description of S. R. P. Apparatus.—The two essential parts of the short range projection method are: (1) the light source; and (2) the translucent screen to receive the striae shadow patterns.

(1) **The Light Source.**—As previously stated, light conditions for revealing the presence of striae are most favorable when the source is as concentrated as possible. To avoid focusing light rays on a pinhole aperture in an opaque screen, and also the disadvantage both of the wandering of the spot of maximum brightness and of a considerable lack of concentration where an ordinary electric arc is employed directly without a lens, a small commercial type electric lamp of more recent design and of unusual concentration was used. The specifications of this lamp are as follows: type, motorcycle, trade name, Edison Mazda 131; con-

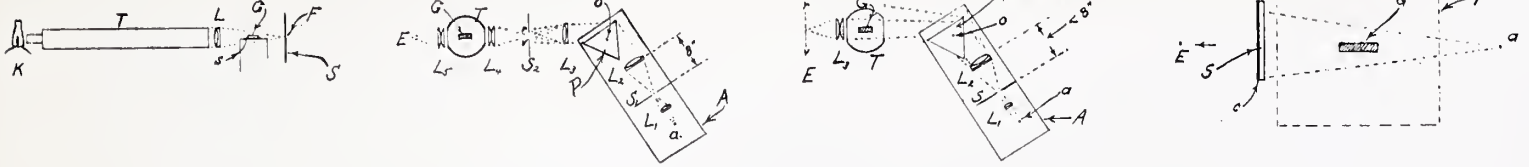


FIG. 1.

(*a*)

Converging Light, Direct View Method of Brashear, employing telescope.

(Vertical longitudinal section.)

K—kerosene flame.

T—4½ ft. telescope with 4 in. objective.

L—achromatic objective, 4 in. diam. and focus about 20 in.

G—glass sample.

s—stop for sample.

S—cross-section of opaque screen placed perpendicular to line of sight.

F—focus of rays at edge of screen, also eye position.

(*b*)

Unmodified Tank Immersion Method, Bureau of Standards.

(Horizontal long. sec.)

a—16-v. lamp source, inverted "U" filament.

L_1—achromat, about 2 in. diam. and 2 in. focus.

S_1—screen with pinhole permitting a short segment of image of "U" filament to act as "point source."

L_2—achromat, 5 in. diam., 8 in. focus.

P—carbon bisulphide prism.

A—support for rotating the parts above named as unit, with axis as at point *o*.

L_3—achromat, same dimensions as L_2.

S_2—screen with slit ⊥ plane of paper.

r and *v*—limits of visible spectrum on screen S_2.

L_4, L_5—converging lenses, 5 in. diam. and 8 in. focus.

I—immersion tank with plate glass windows.

G—glass sample.

E—eye position.

(*c*)

Modified Tank Immersion Method, Bureau of Standards.

(Horizontal long. sec.)

Note—the parts mounted as unit on support *A* are the same and have the same relations here as in Fig. 1 (b), except that the distance from S_1 to L_2 is slightly less than the focal lenth of L_2, so that the beam is somewhat divergent from L_2 to L_3.

T—tank.

G—glass sample.

L_3—uncorrected converging lens, 5 in. diam., 8 in. focus.

E—eye position at focus of spectrum.

r and *v*—limits of visible spectrum. (Not all of spectrum was focussed at *E* at one time. The segment present at one time depended on the angular position of support *A*.)

(*d*)

Short Range Projection Method (Horizontal sec. in line of sight.)

a— 6- to 8-v. lamp, concentrated filament (see separate description).

T—position of tank in immersion test on samples having ground surfaces.

G—glass sample.

S—translucent screen.

b—crystal glass of screens.

c—opal backing of screen *S*.

E—eye position.

DESCRIPTIVE.

Test series.	Apparatus.	Method No.	Type.	State of samples with each method.	Remarks.
1.....	Brashear (Fig. *a*)	I	Direct view	Polished	
2.....	B. S. tank immersion (Fig. *c*)	II	Direct view	Polished	
3.....	Short range projection (Fig. *d*)	III	Projection	Polished	Without tank. Lower light intensity (see discussion).
(3*a*)...	S. R. P. (Fig. *d*)	III	Projection	Polished	Without tank. Light intensity increased from that in Series 3. Series incomplete owing to most of the samples having just previously been ground on the polished ends.
4.....	S. R. P.	IV	Projection	Ground	With tank. Increased light intensity as with Series (3*a*).
5.....	B. S. T. I. (Fig. *c*)	V	Direct view	Ground	

tact, single; volts, 6–8 amps., 3.5; c. p., 28; type of filament, "C" (see Fig. 2 and description below); diam. of spherical bulb, 1 1/9 ins.

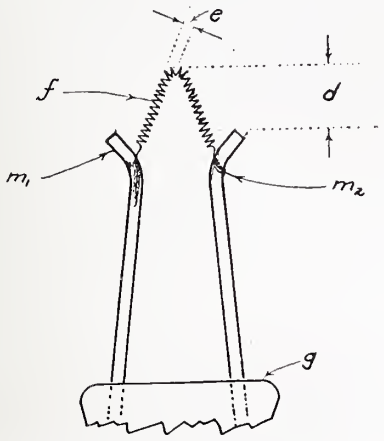


Fig. 2.—Concentrated filament. Axis of beam in plane of paper.
f—tungsten filament soldered to metal supports, m_1, m_2.
d—effective length (about 3/32 in.) of filament as lamp was used in S. R. P. method.
e—width of helix about 1 mm.
g—glass support.

Filament.—The type "C" filament is of tungsten wire in the form of a helix of about 1 mm. outside diameter (Fig. 2). The helix itself is bent into a V-shape, with vertex pointing away from the base of the lamp. The lamp was adapted base downward with the plane of the V parallel to the line of sight, or the direction including source, glass sample, and translucent screen. Thus the filament afforded essentially a very short "line source," with some width (about 1/2 mm.), and of about 3/32 inch length. While this filament provided a source of by no means so great concentration as that of a luminous pinhole, the results obtained with it in the present tests are favorable to the adoption of an electric lamp, with its advantages in use, for striae testing, or wherever light source concentration of moderate intensity is desired. The present results justify further concentration of

filament with the maintenance of good light intensity, in developmental work on such lamps, for "point source" purposes.

(2) **The Translucent Screen.**—While a thin sheet of any light-colored translucent material that diffuses light well enough to prevent direct vision of the source through it, will show the striae shadow patterns, the visibility as far as the screen itself affects it, is a maximum when the translucent material is perfectly free from visible structure, or grain. Otherwise the finer striae, especially if they are very limited in extent, may be partially or wholly obscured, depending upon the nature and coarseness of this structure in the screen. If it were not for its grain a sheet of ordinary white paper would serve the purpose even of the most precise inspection.

Ground glass is objectionable on the same grounds of showing grain, either of line or of strictly granular type according to the manner of grinding. Whether a sheet of glass can be so finely ground as to eliminate serious visible markings on the ground surface and still maintain enough diffusing power to form sharp shadow patterns of the finer striae remains to be determined.

For the short range projection method in the present tests a very satisfactory translucent screen was fortunately obtained[1] that appears to meet the conditions. Indeed, it is not certain that this screen could be materially improved upon for the purpose. Its only possible defect as a screen for the S. R. P. method is in the minor question, possessing some uncertainty, of the desirability of absolute whiteness in such a screen. It was in the form of a plate of crystal glass with opal backing. In dimensions the crystal glass was approximately $1\frac{5}{6} \times 2\frac{1}{2}$ inches, and 2 mm. thick, while the opal backing ran about $\frac{1}{4}$ mm. in thickness. There was no visible structure in the opal glass. This screen was adapted in the S. R. P. method with the opal backing always nearest the eye of the observer.

Dimensions and Procedure, S. R. P. Apparatus.—In the present tests with glass samples having polished surfaces, a given sample was laid on a table top at a distance of about 6 or 8 inches from the light source and on a level with it, and the screen was

[1] This screen was placed at the disposal of the Bureau by the Consolidated Lamp and Glass Co., of Coraopolis, Pa., through the courtesy of Mr. Sargent.

held in the hand, with its edge supported on the table, at a distance of from nothing up to 6 or 8 inches at the most. In the tests with the tank in place (Series 4) the screen was fixed in position in a sheet of cardboard that covered the plate glass window of the tank opposite the source, and the distance from sample to screen was about $4^1/_2$ inches (to center of sample), while the distance from source to sample was about the same as before.

It is to be noted that in both series (3 and 4) where the S. R. P. method was used, the striae shadow patterns were formed with white light, for it was desired to test the method without the use of prisms or lenses. Liquid filters, to make the light from the electric lamp approximately monochromatic, appear to be undesirable because of their serious reduction of the intensity, although the possible use of some sort of color screen with this method doubtless merits further investigation. A satisfactory color screen would of course add to the value of the method.

Description of Samples.

The set of 117 samples were of borosilicate crown glass, of approximate index 1.51 to 1.52, made by a commercial optical glass plant for the government's war work. They were the property of the Ordnance Department and were on hand at the Brashear plant at the conclusion of the war work there. By permission this set of samples has been retained by the Bureau of Standards as its own property for further work with them. They were of rectangular prism shape and uniform size, $1.5 \times 1.5 \times 6.5$ cm., with ends polished and side surfaces in the ground state following the trimming down by Brashear's from the original blank as molded by the glass company. In size this original blank was about $2.5 \times 2.5 \times 7$ cm. From the rectangular prisms, such as comprise this test set, as trimmed down from the original blanks the working into the finished rotating prism form was done, chiefly by making the ends of the rectangular prisms oblique and polishing same. Brashear's tested the samples in the rectangular prism form, and it was in this form that the set of 117 was used throughout the five comparison series of the present work.

Experimental Procedure.

In all five series the samples were viewed through the ends, or lengthwise. The ground side surfaces remained unaltered throughout the tests. Judgment as to the grade of each sample in the set was based entirely on the visibility of the striae under these conditions. Samples in which striae could be distinctly seen were not placed in the first grade. The second grade samples contain lighter striae, and the X glass either heavy or numerous light ones. The percentage of X glass (glass rejected as not usable) was used as a relative measure of the "strictness of the grading."

Observer A, Brashear's regular inspector, first graded a number of samples with Method I, taking them as they came from the boxes in which they had been stored. He separated them into three grades, based entirely, in this case, on visible striae, which he designated as "best," or "good," "fair," and "bad." The Bureau inspectors subsequently throughout the tests used the same grading scheme, which corresponds to the Bureau's customary grading, of A and B, C, and X, or rejected, glass.[1]

When observer A had thus graded a number of the blanks regarded as sufficient to form the desired set for the comparative tests, the samples were numbered and A's grading recorded. They were then graded by B, being placed in position in the apparatus for him one at a time, without regard to numerical order, except to have each sample selected from the lot in a random fashion, and with the number of any given sample unknown to him. This plan was followed throughout the tests. Thus the observer could not be biased by his associating the

[1] During the use of the modified tank immersion method for routine grading by the Bureau as to visible striae, no glass was graded A, and only that which appeared to be free from striae was graded B, where formerly glass appearing free from striae was graded A, and that having very faint striae B. This greater strictness had been adopted because of some question as to the effectiveness of the Bureau's immersion method in its modified form, a question of the monochromatism of the light. Since this question was raised no glass tested by this method was graded A. The results of the present work, however, tend to restore confidence in the Bureau's immersion method as modified, which has been in use more recently and up to the present time.

number of a given sample with a previous grading. The only way in which his memory might have entered as a disturbing factor was through recognition of a given sample by the appearance of its striae. But even in this event the effect on the results could not have been serious, as is shown in discussion of this point later.

In the testing of each sample, where the glass was not unquestionably of grade X, it was rotated, if necessary, through at least 90° (with the long axis kept in the line of sight) to avoid error from a certain amount of non-uniform and hence inadequate visibility with the sample in a fixed position. Such non-uniformity was due to the straight edge of the opaque screen at the position of the eye (see description of apparatus). Also when necessary the sample was turned end for end.

When observer B had finished the set of 117 samples—still with Method I in use—it was similarly tested and graded by observer C. The same procedure, but with only the two Bureau observers, B and C, was followed throughout the remaining four series of tests.

Data and Results.

The data and results are assembled in the following tables. Table 1 gives the gradings, Table 2 the totals and percentages, and Table 3 a comparison of the five methods on the basis of the strictness of grading by the Bureau's inspectors.

Discussion.

The data and results as summed up in Table 3 and the diagram of Fig. 3, require comment other than the statements, based upon obvious comparisons, to be found in the conclusions.

Examination of the data in Table 1 shows that observer B graded the same 54 of the set of 117 samples X in all five series, while observer C's corresponding number was 46. The relation between these two numbers is in good correspondence with that between the two observers' gradings throughout the tests. Of the set, the same 40 of the X's were so graded in all five series by both of the observers in common. Of the remaining samples of the set the same 14 that were graded X in all five series by observer B were not so graded in all the series by observer C,

TABLE I.

Data from Comparison Tests for Striae on 117 Samples with Eleven Complete Gradings.

Series	1.						2.				3.				(3a).				4.				5.			
Observer	A		B		C		B		C		B		C		B		C		B		C		B		C	
Grading	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X
Sample.																										
1		x			x			x			x			x						x			x			x
2	x			x			x			x		x	x			x			x			x		x	x	
3		x			x			x	x			x		x		x				x			x			x
4	x			x			x		x			x		x						x	x			x		x
5		x			x			x			x			x						x			x			x
6		x			x			x			x			x						x			x			x
7		x			x			x			x			x						x			x			x
8		x			x			x			x			x						x			x			x
9		x			x			x		x				x				x			x			x		x
10	x			x				x	x		x			x		x				x			x	x		
1			x		x			x			x			x						x			x			x
2		x			x			x			x			x						x			x			x
3		x			x			x			x			x						x			x			x
4		x			x			x			x		x			x				x			x	x		
5	x				x		x		x			x		x						x			x			x
6		x			x		x					x								x	x			x		x
7	x		x			x		x		x			x			x				x	x			x	x	
8	x			x		x		x		x			x							x		x	x			
9	x			x		x			x			x	x			x				x	x		x			
20	x		x			x			x			x								x			x	x		
21		x			x			x			x			x						x			x			x
2		x			x			x		x				x						x			x	x		
3		x			x			x			x			x						x			x			x
4		x			x			x			x			x						x			x			x
5		x			x			x			x			x		x		x		x			x			x
6		x			x			x			x			x						x			x			x
7	x				x			x			x			x		x				x			x			x
8	x			x		x		x		x		x				x		x			x	x		x		
9	x			x		x		x			x			x					x			x			x	
30	x				x		x				x			x						x			x			x

TABLE 1—(*Continued*).

Series	1.										2.				3.				(3a).				4.				5.			
Observer	A				B				C		B		C		B		C		B		C		B		C		B		C	
Grading	1	2	X		1	2	X		1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X
Sample.																																
31		x				x				x		x		x		x		x						x				x		x
2		x				x				x		x		x		x		x						x				x		x
3		x				x				x		x		x		x		x						x				x		x
4	x					x			x			x		x		x	x			x		x		x	x			x	x	
5		x				x				x		x	x			x		x						x				x		x
6	x				x					x		x		x		x	x			x				x		x		x		x
7	x					x				x		x		x	x			x			x			x		x		x		x
8	x					x		x			x			x		x		x				x		x				x		x
9	x					x		x			x			x		x			x			x		x				x	x	
40	x					x			x		x			x			x						x	x				x	x	
41		x					x			x		x		x		x				x				x	x				x	x	
2		x					x			x		x	x			x				x				x					x	x	
3		x					x		x			x		x		x				x				x					x		x
4		x					x			x		x		x		x				x				x					x		x
5		x					x		x		x		x			x	x			x				x	x		x		x		
6		x					x			x		x		x		x					x			x					x		x
7	x			x			x			x		x		x		x					x			x					x		x
8		x					x		x		x			x		x					x			x		x			x		
9		x					x			x		x		x		x					x			x					x		x
50	x						x			x		x		x	x		x			x				x					x		x
51	x						x			x	x		x			x				x				x		x			x		x
2			x				x			x		x		x		x					x			x					x		x
3		x					x			x		x	x			x					x			x	x			x			x
4	x						x		x		x			x	x		x			x			x		x			x			x
5	x			x		x			x			x		x		x				x			x	x				x			x
6	x				x				x			x		x		x					x			x	x				x		x
7		x				x				x		x		x		x				x				x		x			x	x	
8	x				x			x			x			x		x				x				x		x			x	x	
9		x				x				x		x		x	x					x				x					x		x
60	x					x		x				x		x		x					x		x					x		x	

TABLE 1 —(*Continued*).

Series	1.									2.				3.				(3*a*).				4.				5.			
Observer	A			B			C			B		C		B		C		B		C		B		C		B		C	
Grading	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X
Sample.																																	
61	x				x		x				x		x	x			x						x						x	x			
2			x			x			x			x		x			x								x				x				x
3			x			x			x			x		x			x								x				x				x
4	x					x			x			x		x			x								x				x				x
5			x			x			x			x		x			x								x				x				x
6	x					x	x				x			x			x							x					x				x
7	x			x			x			x		x		x			x								x	x			x	x			
8	x				x				x	x		x				x	x				x	x			x	x				x	x		
9	x			x				x		x		x				x	x					x	x			x				x			
70	x					x			x		x	x		x			x							x					x				x
71	x				x		x			x		x					x							x					x	x			
2			x			x			x		x	x					x					x							x				x
3			x	x				x		x		x					x						x				x			x	x		
4	x			x			x				x		x				x							x					x				x
5	x					x		x			x		x				x							x					x				x
6			x			x			x		x		x				x							x					x				x
7			x			x			x		x		x				x							x					x				x
8			x			x			x		x		x				x							x					x				x
9			x			x			x		x		x				x							x					x	x			
80			x			x			x		x		x				x							x					x				x
81		x				x		x			x	x	x			x			x					x					x				x
2	x					x			x		x			x			x							x		x			x				x
3	x					x			x	x				x			x							x	x				x			x	
4			x			x			x		x		x				x							x					x				x
5	x					x		x			x		x				x							x					x				x
6		x				x			x		x		x				x							x					x				x
7	x				x		x			x		x					x							x	x				x				x
8			x			x			x		x		x				x							x					x				x
9			x			x			x		x		x				x							x					x				x
90			x			x			x		x		x				x							x					x				x

TABLE I —(*Continued*).

Series	1.									2.						3.						(3a).						4.						5.					
Observer	A			B			C			B			C			B			C			B			C			B			C			B			C		
Grading	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X	1	2	X
Sample.																																							
91		x			x			x			x			x			x			x								x			x			x			x	
2	x				x			x			x		x				x			x								x			x			x			x	
3	x				x		x				x		x			x			x			x						x			x			x			x	
4	x				x		x				x		x				x			x								x			x		x					
5	x				x			x			x			x			x			x								x			x			x				
6	x			x				x			x			x			x			x								x			x			x			x	
7		x			x			x			x		x				x			x								x			x			x			x	
8		x			x			x			x			x			x	x										x			x		x			x		
9		x			x			x			x			x			x			x								x			x			x			x	
100			x		x			x			x			x			x			x								x			x			x			x	
101			x		x			x			x			x			x			x					x			x			x		x					
2			x		x			x			x			x			x			x			x					x			x			x			x	
3	x			x			x			x				x			x			x			x					x			x			x			x	
4			x		x			x			x			x			x			x								x			x			x			x	
5			x		x			x			x			x			x			x								x			x			x			x	
6		x			x			x			x		x			x			x			x						x			x			x			x	
7			x		x			x			x			x			x			x								x			x			x			x	
8			x		x			x			x			x			x			x								x			x			x			x	
9			x		x			x			x			x			x			x								x			x			x			x	
110		x			x			x			x			x			x			x								x			x			x			x	
111			x		x			x		x			x				x			x								x			x			x			x	
12			x		x			x			x			x			x			x								x			x			x				
13		x			x		x				x			x			x			x								x			x			x				
14	x			x				x		x			x				x			x								x			x			x				
15	x				x			x			x			x			x			x								x			x			x			x	
16		x			x		x				x		x				x			x								x			x			x				
117			x		x			x			x			x			x			x								x			x			x			x	
Totals	48	26	43	11	21	85	20	26	71	20	12	85	36	5	76	17	5	95	17	19	81	. .	1	36	2	1	5	14	18	85	30	11	76	10	11	96	34	2	81

TABLE 2.

Data and Results, Comparison Tests for Striae in Optical Glass.

Series.	Observer.[1]	No. of pieces first grade.	No. of pieces second grade.	No. of pieces X grade (x).	Usable glass ($117-x$).	Per cent.				
						First grade.	Second grade.	Sum (Usable glass).	X Grade (Strictness).[2]	Average strictness for two observers.
1	A	48	26	43	74	41.02	22.22	63.24	36.76[3]	66.6
	B	11	21	85	32	9.40	17.94	27.34	72.66	
	C	20	26	71	46	17.09	22.22	39.31	60.69	
2	B	20	12	85	32	17.09	10.25	27.34	72.66	68.8
	C	36	5	76	41	30.77	4.27	35.04	64.96	
3	B	17	5	95	22	14.53	4.27	18.80	81.20	75.2
	C	17	19	81	36	14.53	16.24	30.77	69.23	
4	B	14	18	85	32	11.96	15.38	27.34	72.66	68.8
	C	30	11	76	41	24.78	9.40	34.18	64.96	
5	B	10	11	96	21	8.54	9.40	17.94	82.06	75.6
	C	34	2	81	36	29.05	1.70	30.75	69.25	

[1] A. Hageman, Brashear's regular inspector; B. Hinkel, Bureau of Standards regular inspector; C. Dodd.

[2] This number taken as a relative measure of strictness of grading.

[3] To avoid possible misunderstanding due to the grading strictness number of observer A being considerably below the numbers of the other two observers, the point, mentioned in the discussion, should be noted, as to Brashear's relying upon the final test of performance of the finished prisms.

TABLE 3.

Comparison Methods, Bureau of Standards Grading—Summary.

Series.	Method.	Light[1] conditions.	State of samples.	Strictness of grading. Observer. B.	Strictness of grading. Observer. C.	Average.	Date of tests, 1919.
1.....	Brashear	White	Ends polished	72.66	60.69	66.6	July 7
2.....	Tank immersion (B. S.)	Monochr.(?)	Ends polished	72.66	64.96	68.8	July 8, 9
3.....	Short range projection (B. S.)	White	Ends polished	81.20	69.23	75.2	July 10
4.....	S. R. P. + T. I. (B. S.)	White	Ends ground	72.66	64.96	69.2	July 16, 17
5.....	T. I. (B. S.)	Monochr.(?)	Ends ground	82.06	69.25	75.6	July 18, 19

[1] See also description of apparatus.

while the same 6 samples not graded X in the five series by observer B were so graded in all series by observer C. None were graded either firsts or seconds by B in all five series, while C gave first grade to three samples (17, 61, 67), and second grade to none, in all series.

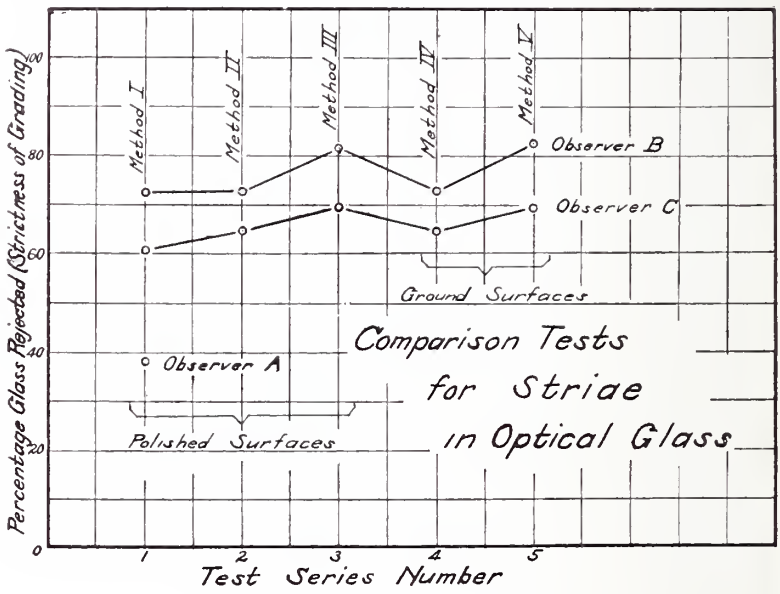


Fig. 3.

The variation (see Fig. 3) from one series to another, of the mean grading strictness number, is the same as that shown by the strictness numbers for each of the individual observers. This parallelism, throughout all the five series, of the two observers' independent judgments as they apply to the set of samples as a whole, tends to build up confidence in the data, for it indicates that a given experienced inspector's grading is consistent.

The fact that observer C's grading strictness numbers throughout the five series of tests are considerably lower than observer B's, is doubtless due principally to a difference in the grading scales of the two individuals. A grading scale is in the last analysis a matter of the individual's judgment, however he may

be called upon to modify that scale to conform to particular requirements. There may possibly enter also in the present gradings by the Bureau inspectors a factor, which is at any rate of minor importance, due to the fact that observer C, while he had had considerable experience in routine testing of glass samples some months previous to the present tests, had not been doing such regular inspection during the interim, and for that reason the faintest striae seen by observer B may have escaped him, owing to his being "out of practice" in detecting the faintest striae. Locating the faintest striae presents the same difficulty as locating a bird in flight when its course has not been previously followed and it is barely visible in the distance. Observer B, on the contrary, had been doing almost daily routine inspection for about two years, up to the time of these tests, and during this period he must have graded tens of thousands of samples. He should thus be expected to show more proficiency in discovering faint striae than observer C. Or a consistent difference in two observers' gradings might be due in part to a difference in vision. This difference in grading does not however affect the conclusions, for it is consistent and thus indicates freedom from erratic judgment on the part of both observers.

Since the ends of the prisms were polished, it was not necessary to use the tank test for Series 2, except for a comparison of the effectiveness of the light conditions in it with the other methods. It did, however, make possible the seeing of striae through the ground sides, impossible in the other two methods where immersion is not used with them. But in these comparative tests the samples were not graded on the basis of striae visible through the sides, but only striae visible through the ends. Thus one sample in particular was noted where striae not visible at all though the ends were plainly visible through the sides. There is nothing to prevent the use, if desired, of a tank with the apparatus of Method I.

During the tests, just before the samples were subjected to end grinding to remove the polished surfaces preliminary to Series 4 and 5, it was learned that the small electric lamp could be run safely at a considerably higher intensity than had been used in Series 3, with correspondingly improved visibility of the striae.

The lamp under these conditions was probably running nearer its rated capacity than with Series 3. (As resistance in series with the lamps four and five 12–16 volt lamps were used in parallel for the two intensities respectively.) This improved visibility of striae with increased intensity of the light means that the percentage, or grading strictness numbers, for Series 3 should in reality be higher than those given. But since the difference would be in favor of the S. R. P. method rather than derogatory to it, the numbers as first obtained in that series with light intensity lower than necessary for safety to the lamp, are retained and presented in the table. The higher intensity was used for Series 4.

To make sure of this increased visibility with the higher light intensity the samples graded in Series 3 as "usable," were re-graded under the same conditions, but with the increased intensity, with results of observer B given in Series (3*a*). Eight of these samples selected at random were then regarded by observer C, with results shown in the same series.

Another factor, besides lower intensity tending to keep the values of Series 3 down, and also those of Series 4, was the real image of the filament formed by the bulb and located about 2 mm. to one side of the filament itself. This real image had far less intensity than the filament, but nevertheless it formed a separate source with intensity sufficient to give, with a slit (pin-hole camera principle), a distinct line of light parallel to that due to the filament itself and some distance from it. Thus there was present with the S. R. P. method as used in these series, the defect of a source extended unduly on account of this real image not coinciding with the filament. This defect is doubtless removable in such lamps, by better centering of the filament in the bulb.

In Series 1, the fact that observer A (Brashear's inspector), is so decidedly below observers B and C in strictness of grading, does not mean that he failed to see the striae. Such an outcome is to be expected since Brashear's test the samples later as to optical performance of the finished pieces, and have found that samples which look poor in the striae test may give good performance in the finished pieces, while samples that appear to be even free from striae (viewed lengthwise) may give unsatisfactory

performance. Upon this fact both McDowell and Hageman of Brashear's laid emphasis. The Bureau of Standards grading, as it has been done at the glass factory in Pittsburgh, has been entirely on the basis of visible striae.

With reference to Series 2, the strictness number of observer B here is the same as his number in Series 1. But there is a marked increase in C's number, and the conclusion is that Method II is at least as effective as Method I.

The general increase of the grading strictness numbers over the first three series is significant. If all experimental conditions for each of the three series had by chance been equal, some increase from one series to the next might have been expected, on the ground that the observer becomes, with each successive grading, better acquainted with the individual samples. But in the present case this explanation rests on only a very slight foundation. The number of samples, 117 in all, is too large for any considerable number of them to be definitely recalled, particularly when the total number of different tests is limited to only five, as in the present work. Furthermore, it is probable that the samples most likely to be recalled are the ones with the heavier striae, samples that were rejected in all the series of gradings. Again, the order in which the samples were placed before the observer was independent in the different tests, and the observer was unaware of the number of any particular sample before him. This explanation is therefore rejected.

The alternative then is that the reason for the progressively increasing strictness of grading for the three successive tests with polished surfaces, lies in the experimental conditions. If this explanation is accepted the conclusion is that the tank immersion test as used for routine testing by the Bureau of Standards, is fully as favorable to strict gradings as the Brashear apparatus in its present manner of operation, and that the S. R. P. method with polished surfaces is at least the equal (see Series 5 and later discussion) of the B. S. modified tank immersion method.

It was not anticipated that Method II would prove itself superior, or even equal, to Method I in effectiveness. In fact it had been rather regarded as somewhat inferior, because of the

question, already mentioned, of lack of monochromatism, due both to the use of uncorrected lenses and to a segment of the spectrum of some length entering the pupil of the eye (see description of apparatus).

It might conceivably be urged that the grading by the Bureau's inspectors, but with the apparatus of Method I, is not so strict as it should be due to lack of familiarity with that apparatus. But Method I belongs to the direct view type, the same as the tank immersion method of the Bureau, and although the light in the former is slightly converging as it traverses the sample while it is slightly diverging in the latter method, the visibility conditions would not appear to be different in the two cases.

Comparing Series 2 and 5—since in Series 5 with ground surfaces the match between glass and liquid may be regarded as perfect, as far as absence of obscuration of the striae by the surface markings was concerned, the higher strictness of the grading for this series as compared with Series 2, is noteworthy. As far as known, the conditions for both series were the same with the sole exception of the necessity for a match of indices between glass and liquid to render the ground surfaces invisible in Series 5. If the match was not perfect then the grading strictness for Series 5 should be lower than that for Series 2, whereas the opposite is the case. If the match was practically perfect for the purpose, as it doubtless was, the two series should be equal as to grading.

A possible explanation to be offered for the difference is one previously suggested for the first three series, *viz.*, a progressively increasing strictness of judgment from series to series in the order in which the tests were actually run (which is the order indicated by the series numbers), an increasing strictness due possibly to constantly improving acquaintance with the samples, or conceivably to some other reason of a psychological nature. But, as already pointed out, memory of the appearance of the striae patterns of individual samples would be expected to be confined almost entirely to those samples showing heavier striae, which in all tests would be rejected anyway; the finer striae were often too numerous to give the individual sample a characteristic striae pattern easily to be recognized. That the increase, in the case of these two series, has a definite cause with an independent

effect on both observers is shown in that each of the observers gives an increase independently.

It is not improbable that some experimental condition, such as adjustment of the lamp filament image on the pinhole, or intensity of the light due to differing voltage on the power circuit used, was more favorable to visibility of the striae in Series 5 than in Series 2. For the factor of light intensity appears to be important to the visibility, over a certain range of intensities at any rate. However, since the illumination system regularly used in the tank immersion test of the Bureau was employed in the present work only for these two series, the cause of this difference in the values, if it was to be found in this system, affects these two series alone. Allowance has been made for this difference, in the manner of statement of the conclusions. For purposes of a strict comparison of the B. S. tank immersion method with the others in the present work, the higher values of Series 5 should be retained.

In any event, with further reference to the difference between the two Series 2 and 5, the comparison of the short range projection method with the others is favorable to its adoption for regular use in the inspection of optical glass for striae, even with white light, which makes possible the elimination of all prisms and lenses from the testing apparatus (see concl. 9).

Brashear's tested the samples in the rectangular prism form, for striae, strain, coma, and other defects such as seed (bubbles). As a matter of fact it does not appear to be necessary to cut glass down from the original molded blank in order to test for strain, as proper annealing should have eliminated this defect from the blank, which could have been tested for strain in the blank form. Further, inspection for striae could have preceded the annealing. Coma as detected by Brashear's in part of the rectangular prism samples is due to the presence of striae, which affect the light rays in the same manner as a lens off axis. According to Brashear's coma will sometimes be found when no striae are visible in the rectangular prism samples as tested endwise at that plant. In such cases, however, an immersion test through the ground side surfaces would doubtless reveal striae.

Because the plane-parallel ends only of the rectangular prisms

were polished, Brashear's did not test them through the sides, with their striae apparatus. Striae inspection, or tests of other kinds, through the ends of the samples in the rectangular prism shape would probably be entirely reliable for judging the final performance of the finished piece if the rays of light passed through the glass parallel to the long axis, as they do, roughly at least when the striae test is made (the light converges somewhat in the Brashear apparatus, Fig. 1*a*). But in the finished rotating prism form the rays do not travel roughly parallel to this axis. Consequently, where the samples have been tested in the rectangular prism shape through the ends only, the performance of the finished rotating prism is the only final criterion of the optical value of the piece, as far as striae affects it, for the purposes desired.

Brashear's have doubtless been willing to permit the limitations of end view only of the samples in the striae test, because of the fact that a sample which shows itself nearly or entirely free from striae (when viewed endwise) may give poor performance in the finished piece, while a sample showing striae to an apparently serious extent may ultimately give good performance. This fact must depend upon the nature, *i. e.*, shape, size, and orientation relative to the rectangular prism axis, of the striae.

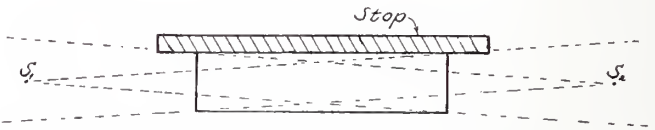


FIG. 4.—Sample in place in apparatus of Method I. (Plan.) (Angles are exaggerated.)

S_1, and S_2,—relative positions of source in direct and reverse positions of the sample, respectively.

While this final test is adequate the method is wasteful where prisms in the finished form have to be rejected, on account of unsatisfactory performance, after the expense of working the glass. The original molded samples should be and can be thoroughly tested for both strain and striae before any trimming is done, so that there will be no necessity of rejecting any pieces in the finished form. Such thorough testing of the original

molded blanks should have been done before the glass reached the lens grinders.

The experience of the Bureau has been that if glass is molded by a suitable method, the molded surfaces will be in such a state of comparative smoothness that the samples can be readily tested for striae by the immersion method without being ground or trimmed at all.

While it is true, as Brashear's experience has proven, that samples may show striae quite decidedly and still give good performance in the finished pieces, it is more economical to reject at the outset all glass showing striae to any serious extent. Furthermore, the tests made of the molded blanks at the outset should be through the sides as well as the ends.

Since the light in the Brashear apparatus is converging while the long axis of the sample has a fixed position, not all the sample is visible, for the parts near the sides are inaccessible to the rays, as Fig. 4 shows, even though the sample is turned end for end. This slight disadvantage can be easily remedied by permitting the prism freedom of movement on its supporting platform, so that by a small amount of horizontal rotation the light can be let through the sample parallel to one side at a time.

This circumstance in Method I of the sample being in a fixed position relative to its long axis may have been responsible for some lowering of the grading strictness values for this method below what they might otherwise be. But the conclusions will not be affected, since they refer to Methods I and II as these had been in regular use. But it is to be noted that the grading values for the S. R. P. method (Series 3 and 4) are doubtless lower than they should be owing to the real image of the filament which has been mentioned. It has already been mentioned that a higher light intensity in Series 3 would have raised the values.

Given a light source of good concentration, any extension of it will lower the visibility of the striae. As stated in the introduction to the paper since a line source is concentrated in one direction, that transverse to its own length, and has maximum extension in the direction perpendicular to this, the visibility of striae with such a source is non-uniform, being a maximum relative to the direction in which the light is most concentrated.

Method I, while using the principle of a really concentrated, or point, source, still gives the non-uniform visibility similar to that due to a line source, on account of the straight-edged, opaque screen at the position of the observer's eye. Thus it is Brashear's custom, when viewing the rectangular prism sample endwise, as is necessary where only the ends are polished, to turn it, at least from the position where side "*a*" (see Fig. 5) is in the downward direction, to that where side "*b*" is down, a rotation of 90°. If necessary for the grading, the sample is placed successively in the eight different possible positions, where sides *a*, *b*, *c*, and *d* are down and the sample is turned end for end. This procedure, of turning the sample from the position of side "*a*" down to side "*b*" down is necessary to compensate for the lowered visibility, with a given position of the sample, of striae components that run perpendicularly to the direction of the screen's edge. In using Method I the Bureau's inspectors noted this marked dependence of position of sample on the visibility and appearance of striae.

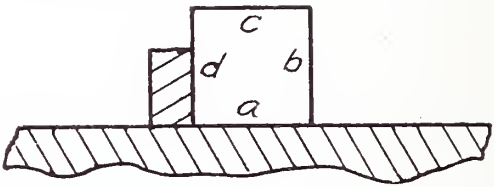


FIG. 5.—Sample in apparatus of Method I. (End view.)

The possible point might be made that since the variation in the percentages do not involve large numbers of individual samples, such variation is easily subject to over-emphasis. But special attention was paid to making all the inspection and gradings in these series with care and deliberateness. Trustworthiness of the results is moreover indicated by the parallelism of the two observers' judgments throughout the tests.

It might be contended that a greater strictness in the grading with a method possessing some novelty is to be expected on psychological grounds, *viz.*, anxiety to see the new method succeed, so that the observer's judgment is affected in a way to enable the method to show up well in comparative tests. But a special effort was made throughout the tests to guard against anything

of this sort, and it is believed the effort has been effective, if the tendency mentioned was present at all in any serious degree.

Advantages of the S. R. P. Method.

There are certain advantages of the S. R. P. method for detecting striae, some of them peculiar to it, which are obvious.

1. The distance from the observer's eye to the translucent screen may always be that of most distinct vision (about 25 cm.), so that the striae shadow patterns have maximum clearness as far as the eye itself contributes to it.

2. Since the striae patterns are all in one plane there is no time lost adapting the eye focus to different depths in the sample, with ensuing fatigue of the observer due to this cause.

3. Striae can be readily located in the glass sample incidental to the turning of the sample during inspection. Such location of striae is necessary where samples are to be trimmed by sawing, by splitting or chipping with the hammer, or otherwise. The relative movements of the shadow figures on the screen determine the positions in the sample of the striae or other defects causing them. This ease of location has also been clearly shown where the rectangular slabs of optical glass as molded by the Bureau with the punty[1] have been tested through the edges by the S. R. P. method. A simple rotation of the slab in its own plane suffices to locate quickly the striae in it.

4. By the use of divergent rays the striae shadow figures are given some magnification on the screen, and this is an advantage up to a certain limit of magnification, which limit is easily avoided with the S. R. P. method. This objection of too high magnification and thus a serious lowering of the visibility is likely to be present where the projection is not of the short range type. (The relative effectiveness of the short range projection method with parallel or convergent light, as compared with divergent, remains to be demonstrated.)

5. The S. R. P. apparatus is compact, and manipulation of the glass samples by the observer can be done with the greatest ease.

[1] The punty method was introduced into the government's glass factory by Capt. Harry Fry.

6. A darkened room is unnecessary for the inspection, as a small hood of stereoscope type will keep extraneous light from the observer's eyes. There is no difficulty preventing outside rays from entering the sample, for with polished samples that do not require immersion a small chamber may be adapted to shield them during the test. This chamber may be curtained to permit entrance and withdrawal of the test pieces. With samples requiring immersion the cover of the tank used, attached to the carriage supporting the piece during the test, affords such protection.

Conclusions.

1. The consistency of a given observer's grading, with a set of samples of sufficiently large number to avoid serious duplications of judgment by memory, is indicated in the numerical results by the very similar variations of the two observers' judgments, as expressed in the summary table by percentages (see diagram also). This conclusion is borne out by the additional fact that the ratio of the number of the same samples in the set graded X by observer B in all the five series, to the similar number graded X by observer C, is in agreement with the roughly constant difference in strictness of grading by these two observers throughout the work.

2. Comparing Series 1 and 2—the illumination system in the tank immersion method as regularly used by the Bureau of Standards makes possible a grading fully as strict as with the Brashear apparatus.

3. Comparing Series 1, 2 and 3—the short range projection method, when the samples have polished surfaces, is at least equal (cf. Series 5, also discussion of Series 2 and 5) to either of the other two methods as they have been regularly used.

4. Comparing Series 1, 2 and 4—the short range projection method with samples having ground ends, and with tank immersion but white light, in spite of the poor index match between glass and liquid due to the white light, compares favorably with the Brashear method and the Bureau of Standards tank immersion method, as these have been regularly used.

5. Comparing Series 3 and 4—the short range projection method with samples having ground ends and with tank im-

mersion (B. S.) but increased light intensity (see discussion) is equal in effectiveness to the S. R. P. method alone with samples having polished surfaces but with lower light intensity, in spite of the imperfect index match due to the white light. Striae can be detected even though their images on the translucent screen are superposed on the ground glass shadow patterns.

6. Comparing Series 3 and 5—the inference is that since the short range projection method with samples having polished surfaces but with white light, is about equal in effectiveness to the tank immersion method with samples having ground surfaces and with approximately monochromatic light, the S. R. P. method with monochromatic light of suitable intensity will be found superior, due to absence of dispersion by the striae under such light conditions.

7. Comparing Series 2 and 4—the Bureau of Standards tank immersion method with samples having polished surfaces, and the short range projection method with samples having ground surfaces but with tank immersion and white light, are of about (see discussion of Series 2 and 5) the same effectiveness. The S. R. P. method with ground surfaces and monochromatic light, giving a perfect index match, should therefore be expected to prove the equal or even the superior (see concl. 8), if only on account of the advantages peculiar to short range projection of the tank immersion method as used at present by the Bureau. (Conclusions as drawn from the two series of tests (2 and 4) will be modified in favor of the short range projection method where the inspection is performed, not with ground surfaces, but either with fractured surfaces, as found in glass fresh from the broken pot or recently broken with the hammer, or samples in the form of molded slabs as they are produced at the Bureau of Standards with the punty, which gives fairly smooth molded surfaces. Such fractured or smoothly molded surfaces, as contrasted with rough ground surfaces, have in general a minimum number of markings.)

8. Reference to the diagram shows that the short range projection method with polished samples (Series 3) stands on an equal footing with the B. S. tank immersion method (Series 5) where that method appears to have had better conditions of

visibility (see discussion of Series 2 and 5). Moreover, the results for these two series (3 and 5) give the highest values for strictness of grading in the entire series of comparative tests. It should therefore be noted that in Series 3 the visibility afforded by the S. R. P. method was not at its maximum, owing to the lower intensity of the lamp source as used during the grading in this series. Therefore, the S. R. P. method in its simplest experimental form and with samples having polished surfaces is the superior of any of the other four methods as employed in these comparative tests.

9. In view of the results as a whole, although they were obtained with a lamp that has not reached the limit of practicable concentration of filament, and in which the real image of the filament due to the bulb did not coincide with the filament itself, the comparison of the short range projection method with the others is favorable to its adoption, for regular use in the inspection of optical glass for striae, even with ground surfaces and white light, which makes possible the elimination of all prisms and lenses from the testing apparatus.

In conclusion, the Bureau is indebted to Brashear's for permission to make one series of the comparison tests at that plant, particularly to Mr. J. B. McDowell, superintendent, and Mr. Hageman, both of Brashear's, and to Colonel Scoville of the Department of Ordnance, Pittsburgh Branch, for their interest and assistance. For needed apparatus loaned for use in connection with this work, thanks are extended to Dr. Frank Schlesinger of Allegheny Observatory, and to Professor Harry S. Hower of the Department of Physics, Carnegie Institute of Technology. The writer wishes to express his appreciation of the coöperation of the Bureau men, and of the assistance, throughout the tests, of Mr. Walter J. Hinkel.

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NOTE ON THE LATENT HEAT OF FUSION OF CRISTOBALITE.

By Edward W. Washburn.

The only means at our disposal for estimating the heat of fusion of quartz is through the application of the law of freezing point lowering. In a previous paper[1] the writer employed the freezing point law of the "ideal solution" in order to estimate the latent heats of fusion of lime and magnesia, the assumption being made that these two substances were so much alike that their solutions might be assumed to obey the laws of ideal solutions.

In the present instance this assumption is inadmissible in any of the known phase rule diagrams of binary systems containing SiO_2 and in applying the law we are therefore restricted to "dilute solutions" that is, to solutions in which the mole fraction of silica is large. For this case the same law is applicable but it may be thrown into the more convenient form

$$L_{F_0} = \frac{RT_{F_0}^2}{\Delta t_F} x_1. \qquad (1)$$

In order to apply this equation to the present case, we must therefore make use of a point in the cristobalite liquidus as near as possible to the melting point of cristobalite. The nearest point of this kind which has been determined at all accurately is the point at 1610° C corresponding to the eutectic between cristobalite and sillimanite. According to Rankin and Wright[2] the composition of the melt at this point is 87 per cent SiO_2 and 13 per cent Al_2O_3. This composition gives 0.0808 as the mole fraction of alumina in the melt, if we assume that none of it is combined with the silica. If on the other hand, as is more probably the case, we assume that practically all of the alumina is present in the solution in the form of sillimanite, the above composition gives 0.0882 as the mole fraction of the sillimanite.

[1] *Trans. Am. Ceram. Soc.*, **19**, 195 (1917).

[2] *Amer. J. Sci.*, **39**, 9 (1915).

In order to calculate the molal heat of the fusion, L_{F_0}, of cristobalite we have only to substitute the appropriate values in equation (1) above, where R (= 2 cal.) is the gas constant, T_{F_0} (= 1710° + 273°) is the absolute melting point of cristobalite, Δt_F (= 100°) is the freezing point lowering (*i. e.*, 1710–1610°) and x_1 is the mole fraction of the solute,[1] in this case Al_2O_3 or Al_2SiO_5.

If we assume that the solute is Al_2SiO_5 with the mole fraction 0.0882 we find L_{F_0} = 6950 cal. If we make the less probable assumption that the solute is Al_2O_3 with the mole fraction 0.0808, we find L_{F_0} = 6360. The average of these is 6650 but since the greater weight should be given to the former value we shall write:

$$L_{F_0} = 6800 \text{ cal. per mole}$$

or

$$l_{F_0} = 110 \text{ cal. per gram.}$$

In the above calculations it is assumed that the solute exists in the solution in the form of molecules having molecular weights corresponding to the formulas written. If association occurs the above value for the heat of fusion is too large.

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[1] Cf. p. 172, eq. 48, of Washburn's "Principles of Physical Chemistry," McGraw-Hill Book Company, New York, 1915.

CERAMIC ABSTRACTS.

General.

The efficient arrangement of a clayworks. *Brit. Clayworker*, 28, 182 (1919). —In most plants the output is increased with increased sales and reduced with reduced sales. A better way is to have a standard output with provision made for increased output in the future based upon the past growth. Every plant should have an entering- and leaving-end and the material should pass from one operation to another in as nearly a straight line as possible. The individual output of each machine should be established with extreme care to determine how it will fit up with the other parts of the arrangement. In designing a plant a plan of the works is made to scale, $^3/_8"$–$^1/_2"$ = 1 foot, showing all features of the scheme, and arrangement. Then for each machine an approximate shape to clearance dimensions is cut out in cardboard to the same scale as the plan, showing the position of the drive, the position of the operator, and the extreme travel of the working parts. At the same time cardboard cutouts are made of transfer trucks, bins, work benches, etc. This is done for every piece appearing on the tabulated sheet. The cutouts are now combined to the best advantage so that all may be busy all the time. Each piece should be considered with respect to the other and each should be moved about until the best arrangement has been found. All unnecessary travelling should be avoided and the gradients and differences in level between machines, etc., should be carefully studied. Great care should be taken in handling the finished brick as they are more costly than clay and cheaper transportation may be used for clay as it is not damaged by transport. When the cardboard cutouts representing the buildings and machinery have been arranged they should be tested by the handling factor (weight × distance) to reduce these as much as possible.

H. G. Schurecht.

Manufacture of roofing tile. *Brit. Clayworker*, 28, 179 (1919).—Plain roofing tiles may be made most satisfactorily by "running" them out of a brick machine fitted with a special mouthpiece so as to form a column of clay of the desired width and thickness, which, when cut into pieces the length of the tiles, will provide a clot which is later pressed, or the nibs may be formed by the deft finger of an attendant. Some difficulties and methods of overcoming them are described as follows: (*a*) In one case the clay was "short" and could not be handled without breaking. The clay occurred in stratas of different kinds, some being too high in fine silt. The clays high in fine silt often appear as plastic as those high in clay substance so the "feel" test

cannot always be relied upon. A method of testing for silt in a clay is to stir with water and allow the same to settle. Decant liquid and repeat several times. It will be found that clays high in silt will leave a comparatively large residue in the beaker. Using a clay low in silt overcomes this difficulty. (*b*) Jagged corners were produced on the column of clay. When more water was fed to the corners in lubricating the mouth piece and the shape of the mouth piece was altered this trouble disappeared if highly plastic clays were being used but it was not entirely overcome where silty clays were used. (*c*) The column of clay was curved—due to the clay passing out more rapidly from one side of the mouth piece than the other. This was prevented by enlarging the hinder part of the mouth piece on the inner edge of the curve so as to allow the clay to enter the mouth piece more freely. (*d*) The tile cracked in drying —due to insufficient lubrication of the clay in the mouth piece. This was due to shutting off the water in the lubricator of the mouth piece and the trouble was overcome by keeping the water turned on at all times. (*e*) The top of the tiles were slightly rounded—due to expansion of the tile as they came through the mouth piece. By altering the shape of the die this was overcome. (*f*) Laminations caused by the screw of the auger being too near to the mouth piece were overcome by inserting a barrel between the machine and mouth piece. The mouth piece had a scaled interior composed of a series of overlapping sheets of copper or zinc, so arranged that the water escaped through the overlapping portions. H. G. SCHURECHT.

Cements for spark-plug electrodes. H. F. STALEY. *Report No. 53*, of The National Advisory Committee for Aeronautics, 33–34 (1919).—Considerable trouble has been caused in airplane engine work through the breaking of the central electrode of spark plugs. An investigation of this problem by the Bureau of Standards shows that in many cases the cement used to hold the nickel electrode wire in the porcelain is of such a nature that it rapidly eats away the wire through oxidization, when exposed to the high temperatures of the engine cylinder. A cement composed of silicate of soda and raw kaolin has been found to give the least trouble in this respect. In cases where the cement holds the wire firmly in the porcelain the latter often cracks when subjected to heat—due to the difference of the coefficients in expansion of the wire and the porcelain. The breaking of the porcelain does not seem to be due to leaky plugs as has often been supposed to be the case. On account of the difficulties attending the use of any form of cement between the porcelain and central electrode, the elimination of the cement and the use of a mechanical seal at the top of the porcelain is greatly to be desired. In such a plug only a porcelain strong enough to safely withstand the resulting stresses should be used. The porcelain recently developed by the Bureau of Standards is believed to meet these requirements. H. G. SCHURECHT.

Method of measuring resistance of insulators at high temperatures. F. B. SILSBEE AND R. K. HONAMAN. *Report No. 53*, of The National Advisory

Committee for Aeronautics, 1–13 (1919).—The temperature of the body insulators of a spark plug within a metal shell seldom exceeds 250° C in water-cooled engines. The tip of the inner end, however, may reach temperatures as high as 900°–1000° C. The electrical resistance was therefore studied between 200°–900° C. In starting an engine 6000 volts are necessary but after this only a comparatively low voltage (800) is necessary. The spark lasts about a few thousandths of a second and the interval between the sparks is about 0.1 second so the average voltage is about 150. Most of the work was with cup-shaped specimens similar to the standard test piece No. 1 adopted by the A. S. T. M., except that the side walls were uniform in thickness. The advantages of this type of specimen are as follows: (1) The conduction takes place through the bottom of the cup which has definite and easily measured dimensions; (2) the large area and small thickness of the bottom insure a relatively large current even with material of high resistivity; (3) the path over the rim of the cup for any surface leakage is relatively long; (4) a satisfactory constant can be made between the specimen and the electrodes by immersing the bottom of the cup in a conducting fluid (in these experiments, melted solder) and by inserting some of this fluid inside the cup to form the upper electrode. In cases where cup specimens were not available, measurements were made on assembled spark plugs, and also on spark plug insulators, and on short pieces of tubing. In these cases the conduction took place between a central electrode and either the shell of the spark plug or a band of platinum deposited around the shell of the spark plug. The measurements with this type of specimen definitely indicated the resistance of the specimen, but owing to the uncertainty as to area of contact and the location of the lines of current flow, it was difficult from such data to compute with accuracy the true resistivity of the material. The resistivity was obtained by multiplying the observed resistance by a factor K, which varied with the shape and dimensions of the specimens. For the cup-shaped specimens $K = \frac{\pi}{4}\frac{d^2}{t}$, where d is the diameter of the bottom and t the thickness of the cup. For tubular specimens $K = \frac{2\pi l}{2.30 \log_{10}\frac{R_2}{R_1}}$, where l equals the length of the external conducting band measured parallel to the length of the specimen, and R_2 and R_1 are, respectively, the external and internal radii of the insulator. The most satisfactory method for the rapid comparison of different types of insulating materials is by the use of the volt-ammeter, using alternating current. Under these conditions the observed resistance is substantially independent of the frequency of the voltage and the time of application and the convenient values of 60 cycles and 500 volts were adopted for the work. A bridge method using direct current would be objectionable because of the variable amount of polarization which would occur. Some typical results are as follows:

Material.	Te Value. °C.
Fused silica	890
Best porcelain	790
Mica plug	720
Av. of 3 aviation plugs	650
Av. automobile plugs	490

H. G. Schurecht.

Some new appliances in potting. A. S. W. Odelberg. *Pottery Gaz.*, 509, 1214–1218 (1919).—An electrical conveyor has been installed in the Gustafsberg Pottery, Sweden, for conveying ware from the bisque ovens to the ware-house, from the glost ovens to the ware-house and for transporting from the glost ware-house through the various other ware-houses to the packing shed. This work is now done with 50 per cent less manual labor, with a saving of two hours' time, and with an appreciable reduction of broken ware. For making oval dishes in a jolly, a new method is described in which the jolly is perfectly rigid, all movements being in the jigger head, thus overcoming the disadvantages of the jolly heretofore employed which proved very tiresome to the operator. The grooving of insulators is now accomplished by a felt wheel acting like a circular saw and operated at about 4000 r. p. m. The groove is made in a few seconds by this method. The formation of cup and mug handles in steel dies, heated by electricity, was referred to. Until 1914 platinum wire was used as the heating element, but since then nichrome wire has been substituted and found to last longer and the cost is only a small fraction of that of platinum. The electrical method of handle-making is much quicker and cheaper than the old method of pressing in plaster molds. In the firing of boilers and ovens the "Mono" apparatus, which records the carbon dioxide in the gases as they leave the furnace, effecting economy in fuel and preventing smoking in kilns, is used. A three-color printing machine in which the paper passes through three rollers of exactly the same diameter is described. It is found best to apply yellow with the first roller, pink with the second and blue with the third, as blue is not seriously damaged by a mixture of yellow and pink. H. G. Schurecht.

Efficiency in wall and floor tile production. *Brick Clay Record*, No. 11, 961–964 (1919).—The plant of the Robertson Art Tile Co., Morrisville, Pa., is described. Saggers are made by a hydraulic press and are very satisfactory. The kilns are loaded with portable loading machines of the Brown type. The company has devised its own sand-screening machine which consists of a revolving drum which leads to a cylinder formed of wire, with spacing between the wires sufficient to allow single floor tile to drop through, whereas the tile which stick together work their way to the end of the cylinder. A battery of presses operated by hydraulic power is used for making the tile.

H. G. Schurecht.

Geological.

Magnesite in West Australia. F. R. FELDTMANN. *The Mining Magazine*, **21**, No. 4, pp. 240–242 (October, 1919).—The magnesite deposits are located near Bulong, about 20 miles east of Kalgoorlie, and on the west side of Lake Yindarlgooda. The district is made up of a complex of highly basic rocks, the major portion of which consists of serpentine with which the magnesite invariably is associated. A few small areas of gabbro or amphibolized gabbro and several large porphyritic dykes make up the minor part of this complex of basic greenstones. In and near the magnesite area a few small dykes are found. East of this greenstone mass is a belt of rocks of plastic origin, known as pebble breccia. Still further to the east is a second area of greenstones less basic than the rocks of the magnesite area.

The largest magnesite-bearing serpentine area is located about $2^1/_4$ miles due east of Bulong and covers about 350 acres, the southern portion of this area being much covered with surface deposits. North of this main mass are several smaller occurrences of the magnesite-bearing serpentine. The largest of these covers about 90 acres. It is possible that other areas may occur further to the north near Mt. Taurus.

In the pebble breccias and near the boundary of the serpentine, small lenses of magnesite have been found. According to the author the magnesite in these patches has been dissolved out of the serpentine by surface waters and redeposited in the plastic materials.

The magnesite occurs mainly in the serpentine in the form of short veins which are irregular both as to strike and dip. Most of these veins are only a few inches wide and many of them are threadlike. Where the serpentine is greatly decomposed the veins are very numerous and may be easily quarried. In a few places the magnesite forms a surface deposit which is usually more discolored than the veins. The largest of these surface deposits extends over a large part of an area of about 15 acres.

The proportion of impurities in the magnesite varies considerably as shown by the following analyses:

ANALYSES OF BULONG MAGNESITE.

	1.	2.	3.
Magnesia, MgO	47.36	44.96	44.31
Carbon dioxide, CO_2	51.69	49.33	47.76
Combined water, H_2O+	...	0.08	nil
Moisture, H_2O-	0.15	0.97	1.17
Silica, SiO_2	0.12	1.12	4.99[1]
Alumina, Al_2O_3; Ferric Oxide, Fe_2O_3; Ferrous Oxide, FeO	0.16	0.56	0.42
Lime, CaO	nil	1.06	nil
Sodium chloride, NaCl	0.53	1.76	1.39
Potassium Chloride, KCl	0.01	0.09	0.08
Magnesium Chloride, $MgCl_2$	0.08	nil	0.11
Sulphur Trioxide, SO_3	trace	0.13	0.15
Total	100.10	100.06	100.38

[1] The specimen contained small veins lined with chalcedonic silica.

The deposits were not being regularly worked at the time of Feldtmann's visit. In 1917, 73 tons were shipped from Bulong, the export value of the mineral being estimated at slightly less than £4 per ton. E. D. Elston.

Geology and mineral deposits of a part of Amherst township, Quebec. M. E. Wilson. *Can. Dept. of Mines, Geol. Survey,* Memoir **113,** No. 96, Geological Series, 1919.—Extensive deposits of kaolin occur in the St. Remi district which is in the southern part of Amherst township, Labelle county, Quebec. The district is at the western end of the Huberdeau branch of the Canadian Northern railway and is about 30 miles north of the Ottawa river and almost equidistant from Montreal and Ottawa.

Kaolin has been discovered in two localities, the principle occurrences being in a zone about 1000 feet wide in Grenville quartzite. This deposit extends north-northwest from lot 8 to lot 2, range VI, south, and the kaolin occurs in a much shattered zone, as fine disseminations between the quartz grains, in veins following the planes of fracturing and also in more extensive deposits which in some instances attain a width of 100 feet and a length of several hundred feet. Due to the thick covering of glacial drift the whole extent of the deposit has not been ascertained. Information obtained from trenches, test pits and stripping, however, indicates that the zone is continuous for approximately 7000 feet. The most extensive deposit of kaolin in this shattered zone occurs in lots 5 and 6. Here as shown by stripping, test pits and bore holes, the lead of kaolin is almost continuous for 1400 feet, its width varying from a few feet up to 100 feet, and at one point having a thickness of 150 feet. The kaolin content in masses of kaolin is generally not less than 35 per cent even though the kaolin contains quartzite either as disseminated grains or in fragments. The kaolin leads in the shattered zone are numerous and range from a fraction of an inch up to 65 feet in width.

Farther south, on lot 8, range IV, and near Pike Creek another kaolin deposit occurs. Although no connection has as yet been found it is possible that the deposit may be another outcrop on the same zone as that described above.

Of importance is the fact that the kaolin deposits are associated with a zone of faulting and shattering in the Grenville quartzite. Whether a series of small faults or a single greater fault has brought about the deformation is not clearly discernible. Local evidence would indicate that considerable movement has taken place. Again it is important to note that wherever kaolin is found the quartzite shows much jointing. In addition the quartzite throughout almost the whole of the fracture zone has been much broken and crushed.

According to Wilson, the evidence seems to show that not only is the kaolin due to deposition in openings caused by deformation of the quartzite, but that large masses of kaolin have been formed by replacement of the quartzite. Whether the kaolin owes its origin to superficial weathering and downward percolating waters or to thermal ascending waters has not been determined by the author, since each method seems to be borne out in part by certain lines of evidence.

The kaolin deposits in the northern part of the fracture zone are composed almost entirely of white to cream-white kaolin and quartz while to the south the greater part consists of varying shades of red, brown or yellow due to disseminated hydrous iron oxide. Whether this color persists with depth is unknown. Uncommon impurities consist of fine flakes of muscovite, aggregates of tourmaline and disseminated flakes and aggregates of graphite. The paucity of impurities in the white to cream-white kaolin is shown in the following analyses of the washed product.

Analyses of kaolin from Amherst township:

ANALYSES OF KAOLIN FROM AMHERST TOWNSHIP.

	1.	2.
Silica	46.13	44.43
Alumina	39.45	40.48
Ferric iron / Ferrous iron	0.72	0.04
Lime	none	0.24
Magnesia	none	0.36
Potash	0.20	....
Soda	0.09	....
TiO_2	....	....
Loss on ignition	13.81	14.46
Moisture	....	....
Total	100.40	100.01

According to Wilson, an analysis made by A. G. Spencer shows the composition of the discolored kaolin to be as follows:

	Per cent.
Silica	54.24
Alumina	34.24
Iron oxide	2.04
Lime	2.54
Magnesia	0.46
Loss on ignition	5.87

The materials which occur in the fracture zone are white kaolin, discolored kaolin, kaolinic quartzite, kaolinized granite (cornish stone), and kaolinized garnet gneiss. Owing to the incomplete state of development work any attempt at estimating reserves of these materials is attended with difficulty. Statements of prospective possibilities show the following estimates subject to conditions considered in detail in the paper.

White kaolin—approximately 75,000 tons for every 50 feet of depth; *discolored kaolin*, 250,000 tons; *kaolinic quartzite*, 300,000 tons to 900,000 tons; *cornish stone*, extent unknown; *kaolinized garnet gneiss*, unknown.

Tests made by J. Keele of the Mines Branch are cited and mention is made of the uses of the various kinds of material, as follows:

Kaolin—For the body and glazing of chinaware, porcelain, wall and floor tile, electrical insulators, enamelware, stoneware, etc., filler in manufacture of papers, cotton, and other textiles; as a constituent of certain plasters, paints and coloring agents; as face powder, polishing powder, and in medicinal preparations.

White kaolin and quartzite—Can be mixed with the Pleistocene marine clay of the district to make fire-brick.

Discolored kaolin—Can be used as a fire-clay.

Kaolinic wall rock—For manufacture of ganister type of silica brick, and when freed from kaolin content is suitable for use in manufacture of lime-bonded silica brick, glass, carborundum, and as a steel foundry sand.

Kaolinized garnet gneiss—Possibly as a fire-clay.

Brief notes on the equipment and mining methods and on the production of china clay are also included. E. D. Elston.

Preliminary report on the economic geology of Hazelton district, British Columbia. J. J. O'Neill. *Can. Dept. of Mines, Geol. Survey,* Memoir 110, No. 89, Geological Series, 36–37 (1919).—Mention is made of a clay from Smithers along the Bulkley river, and results of tests on this clay are given showing it to be suitable for making common brick provided 20 to 30% sand is mixed with it to reduce shrinkage. The clays of this district are chiefly gumbo or boulder clay and are of no economic importance.
E. D. Elston.

Sand and gravel in 1918. R. W. Stone. *U. S. Geological Survey, Mineral Resources of the U. S.,* 1918, Part II, 13 (1919).—Tabulated statements concerning the production of glass sand and brief notes concerning glass sands are to be found in this report. E. D. Elston.

Magnesite in 1918. C. G. Yale and R. W. Stone. *U. S. Geological Survey, Mineral Resources of the U. S.,* 1918, Part II, 7 (1919).—The publication contains brief statements concerning the magnesite industry, including statistics of production, general facts concerning domestic and foreign occurrences and production of magnesite. E. D. Elston.

The Harricanaw-Turgeon Basin, Northern Quebec. T. L. Tanton. *Can. Dept. of Mines, Geol. Survey,* Memoir 109, No. 94, Geological Series, 58–59 (1919).—Deposits of clay, some of which are suitable for the manufacture of brick, are briefly described in this publication. E. D. Elston.

Surface deposits of southeastern Saskatchewan. J. Stansfield. *Can. Dept. of Mines, Geol. Survey,* Summary Report 1918, Part C, 43C–44C.

(1919).—Tests were made by J. Keele upon samples of two shales secured near Fairlight, Saskatchewan. According to Keele the shales might be used for manufacture of building brick or for fire-proofing or hollow building blocks. E. D. ELSTON.

The Mackenzie river basin. C. CAMSELL AND W. MALCOLM. *Can. Dept. Mines, Geol. Survey*, Memoir 108, No. 92, Geological Series, 100–103 (1919).—Clay deposits on the Athabaska river and its tributaries and clays and shales along the Grand Trunk Pacific railway are briefly treated and several laboratory tests are given. E. D. ELSTON.

Road materials in the vicinity of Regina, Saskatchewan. L. REINECKE. *Can. Dept. of Mines, Geol. Survey*, Memoir 107, No. 90, Geological Series, 3–4 (1919).—The use of natural burnt clay derived from beds adjacent to burned coal seams is mentioned in connection with road materials. A test proved the material as used alone was unsatisfactory for such a purpose. E. D. ELSTON.

Geology of the disturbed belt of southwestern Alberta. J. S. STEWART. *Can. Dept. of Mines, Geol. Survey*, Memoir 112, No. 93, Geological Series, 61–62 (1919).—Tests made upon shales show materials are of poor quality which after modification might be used for wire-cut bricks or hollow blocks. E. D. ELSTON.

Refractories.

Preparation of raw materials for the manufacture of glass-house pots. B. J. ALLEN. *Brit. Clayworker*, 28, 172–176 (1919).—In testing 21 fire clays of Great Britain which did not meet the requirements of a No. 1 refractory, it was found that 17 could be sufficiently purified by deflocculation to meet these requirements as far as alumina is concerned. For example, a low-grade sagger clay was purified by deflocculation and 30 per cent non-plastic material removed. After treatment this clay when fired to 1200° C was vitrified. The use of deflocculated clay is recommended as it permits a higher percentage of grog, decreases the temperature of vitrification, and reduces cracking in burning. Grog of the same composition as the fire clay should be used. High-fired grog produces the best results as it not only becomes better distributed in the body but it was shown that about 20 per cent of the alkalies are volatilized by exposing to high temperatures. Black-cored grog should never be used as it causes blow-holes and pin-holes in the glass. The grog should be ground in dry pans with the rollers raised one inch from the bottom of the pan. The scrapers should be set to throw a good body of material under the rollers, with the object of crushing the material into angular rather than spherical fragments. When a magnet is used to separate the iron, care should be taken to prevent loss of iron particles when a drop in voltage occurs. For mixing clay with grog the dry pan is not very efficient because it reduces the grain-size of the grog. Pugging is also unsatisfactory as it does not permit

thorough mixing. Blunging the body in slip form is considered the most satisfactory as the mixing is more thorough than that secured by pugging and the grain size is not altered. A 4-foot blunger should be operated at a high speed —about 80 r. p. m. The alkaline water should be warm. The clay is added to the water first, the fine grog next and the coarse grog last. Some clays which are inclined to gel may be improved by adding barium carbonate. The use of sulphite cellulose liquor as a substitute for sodium silicate is helpful in some cases.

Casting a covered pot in a plaster mold is difficult. It is not possible to cast thicker than $^{7}/_{8}$–1 inch by this method. A hot, excessively dry mold does not give good results as this condition gives rise to "filling marks" and causes the cast clay to come away from the mold too soon. For this reason the experienced caster always sponges the face of the mold taken from the drying shed. Casting by a vacuum casing placed around the outside of the mold permits the casting of thicker walls. If coarse grog is used the insides of pots cast by this method are often rough and should be finished by hand—care being taken not to tear out the grog. An abnormally thick pot or a quicker cast may be obtained by applying pressure to the slip inside in addition to the vacuum on the outside. For casting covered pots the mold is constructed in four parts, namely, base, two sides and mouth piece. The arrangement permits the base to be cast thicker than the sides. The excess slip is removed by suction rather than by turning the mold upside down. In using a vacuum pump for casting it is necessary that a constant vacuum be maintained. The steam-jet, air ejectors are not desirable, since the vacuum varies with the steam pressure, but the Leblanc rotary ejector and the Minlees-Leblanc air pumps are very efficient. H. G. SCHURECHT.

Refractory materials used in coke-oven construction. W. C. HANCOCK. *Brit. Clayworker*, **28**, 155–158 (1919).—The special qualities required are (1) resistance to high prolonged heating under normal or increased pressure; (2) resistance to mechanical abrasion; (3) resistance to sudden changes of temperature; (4) impermeability to gases; and (5) resistance to chemical attack, especially by sodium chloride at high temperatures. Fire-clay brick are used in England because the coal which is washed contains 15 per cent water and fire-clay brick are more resistant to moisture than silica brick. In the U. S. coals are not washed and hence contain only 5 per cent moisture and therefore silica brick are used, being superior in heat conductivity to the fire-clay brick. The so-called "fire-clay" brick used in coke oven construction contain a higher per cent of silica than most fire clays and are made by mixing a silicious material like ganister with a high-grade fire clay. In a satisfactory brick the silica content should not be less than 80 per cent, ferric oxide should not exceed 2 per cent, the alkalies should be less than 1.3 per cent, and the lime and magnesia about 0.5 per cent. Regarding texture of brick it was found (1) fine-grained material is generally less refractory and more susceptible to chemical attack than coarser-grained material; (2) coarse-

grained material resists sudden changes of temperature better than fine-grained; (3) grain size obviously affects porosity and permeability; (4) grain size affects heat conductivity; and (5) fine-grained material appears to resist abrasion better than coarse-grained. Expansion tests under heat showed that bodies containing grog had less expansion than the neat clay while increasing grog decreased the expansion. If salt is present in the coal it would readily combine with the free quartz and fire clay. Iron oxide would become volatile from the chlorine and thus penetrate further into the brick. By washing the coal with hot water most of the soluble salts are removed. Tests were made on the fluxing effect of unwashed and washed coals by heating pieces of fire clay in a crucible with 10 grs. of the various coals. This was repeated twice and the clay was washed free from soluble salts. The fire clay contained 1.7 per cent alkali reckoned as K_2O. After the test it contained the following percentages:

	With unwashed coal, per cent K_2O.	With washed coal, per cent K_2O.
Coal No. 1	2.38	1.78
Coal A	3.09	2.46
Coal B	2.20	1.96
Coal C	2.75	2.28

Similar experiment to determine whether moisture favors the action of salts on coal showed that it does as is shown by the following results:

	Dry coal, per cent K_2O.	Moist coal, per cent K_2O.
Coal No. 1	1.77	2.43
Coal A	0.89	1.04
Coal B	0.59	0.68
Coal C	1.04	2.25

H. G. SCHURECHT.

Refractory products with a fixed volume; zircon, refractory mineral and abrasive. M. A. BIGOT. *Ceramique*, **37**, 191–193 (1919).—Refractories used are chiefly (1) silica products; (2) bauxite brick; (3) fire-clay brick; (4) fire clay plus graphite; and (5) magnesite. The volumes of all of the above products change with temperature. The volumes of all but the silica products decrease with increase in temperature while those of the silica products increase with an increase in temperature. A class of refractories whose volumes remain practically constant are as follows: Natural products such as natural corundum, natural zircon, natural graphite and spinels such as the iron-chrome spinel. Artificial products such as artificial corundum, bauxite fused in a cupalo, carborundum, fused quartz and artificial graphite. This investigation was limited to zircon. The fusion point of zircon is above 2000° C. When burned to 1720° C the contraction was 0.55 per cent, the apparent density 2.90, and the porosity 9.0 per cent. A mixture with 3–4 per cent lime produces a product which when burned to 1500° C has a porosity of 4–7 per cent. The fusion point of the mixture is 2000° C. The addition of oxide of

iron lowers the melting point considerably. For instance, a mixture of 83 parts zircon, 5 parts slaked lime and 12 parts iron oxide had a fusion point of 1820° C. The addition of 8 per cent kaolin produced a product having a fusion point of 2000° C. A mixture of equal parts of kaolin and zircon had a fusion point of 1460° C. In this manner it behaves very similarly to glucinium oxide, which melts at 2400° C, while an emerald, which is a mixture of glucinium and kaolin, melted at 1470° C. In slagging tests it was found that zircon was resistant to basic and acid slags, being superior to most refractories in this respect. As an abrasive it may be classed with corundum and carborundum. H. G. SCHURECHT.

Chrome brick. *Brit. Clayworker*, **28**, 178 (1919).—Chrome brick are at the present time in less demand than magnesite brick. This is due largely to the difficulty of obtaining chrome ore of good quality but this difficulty has decreased and the time is not far distant when chromite will be used extensively. Chrome ore has the advantage over silica, clay or magnesite in that it is neutral in character. Chrome ore is one of the chief sources of the metal chromium, now extensively used in alloy steels, and for this reason the ore used for refractories must be of low quality or its cost would be prohibitive. At the Chaltane brick works in Russia these low-grade ores are purified by a secret process. Brick of good quality may be bonded with fire clay or lime. The brick are burned to 1500° C and may be stacked in a kiln to the height of 10 feet. The brick have a cold crushing strength of 1800–5800 lbs. per sq. in. When lime is used as a binder the burning shrinkage is small but when clay is used it is comparatively large. Lime should not be used in quantities over 5 per cent. Lime bonded brick are more resistant to heat than clay bonded brick. The average clay bonded brick has a softening temperature of 1700° C while the lime bonded brick withstands a temperature above 1850° C. The crushing strengths of chrome bricks are higher than those of magnesite bricks. H. G. SCHURECHT.

Iron oxide and silica brick. *Brit. Clayworker*, **28**, 179–180 (1919).—Silica brick containing iron oxide but no lime were soft when burned while those containing lime were hard. A microscopic examination showed that brick free from lime contained little or no tridymite. Those in which lime and iron oxide were used showed considerable tridymite. This is to be expected as tridymite is only formed from silica which has been in solution and in brick free from lime little fusion occurs. The presence of iron oxide did not reduce the refractoriness of the brick very much under oxidizing conditions—even when 30 per cent was used. When heated under reducing conditions the refractoriness was decreased much more, *i. e.*, about four cones for 30 per cent iron oxide. The fluxing action of iron oxide is much greater for silica containing a high per cent of impurities such as potash, soda, lime and magnesia. For example, the refractoriness of a silica brick high in these impurities was decreased two cones upon the addition of one per cent of iron oxide to the

mixture. The iron oxide should also be fairly pure as any impurities present would facilitate fusion. The addition of iron oxide together with lime is recommended when pure silica is used. H. G. SCHURECHT.

Lime and Cement.

The cause of unsoundness in portland cement. Further discussion. B. F. ERDAHL. *Concrete, Cement Mill Section*, **15**, 51 (1919).—This is a further discussion of the subject of unsoundness, following those articles given in previous abstracts (THIS JOURNAL, **2**, 673 and 960). The author takes up the question of the embedding theory, *e. g.*, unsoundness is due to particles of free CaO embedded in fused particles of clinker. All cement clinkers contain free or uncombined lime in the clinker. But $3CaO.Al_2O_3$, $2CaO.SiO_2$, and compounds of Fe, Mn, MgO, and the alkalies exist enclosed in a magma of fused $3CaO.SiO_2$. These compounds tend to hydrate as well as does the free lime when gauged with water. The formation of the clinker is suggestive. The limestone is finely ground and evenly distributed throughout the raw mix prior to entering the kiln, and becomes calcined before reaching the clinkering zone. In the calcined condition the CaO exists as a fine dry powder, whose particles are extremely small, possibly of near-molecular size. In the clinkering zone the CaO particles become fused in the magma and the greater part of the CaO is used in the chemical combinations taking place. The remainder goes into solution or into a segregated viscous state rather than into the form of nuclei. When the clinker is ground some of these particles of near-molecular size will be liberated, but a great many are too minute to be reached through mechanical means and remain embedded in the clinker. This will be the case with all cements both sound and unsound. If the CaO particles hydrate on addition of water, the soft-burned clinker should prove sounder than the hard-burned. The latter, however, is known to be the sounder. If the particles do not hydrate in the "time of set" pat, the investigators who favor the embedding theory assume that hydration will occur in the boiling test. It is doubtful, however, if any more hydration of free CaO takes place at 100° than at 20° C. This can be proven by making a regulation pat of a sound cement, allowing it to set, and then passing it through the boiling test. If it is then ground to a fineness equal to that of the original cement and another pat made, it will set. If it is then boiled it will pass the test. This experiment shows that unhydrated compounds existing in the boiled pat but partly liberated through the second grinding, hydrate under the second gauging and cause a set. These unhydrated materials existing in the original pat consist of both silicate compounds and free CaO. The quickly acting $3CaO.Al_2O_3$ has probably become completely set in the first set. The second set is then likely due to setting of unhydrated silicates. In grinding, the particles of clinker will break along the lines of least resistance formed by the CaO particles, which will thus be released, and hydrated on addition of water. Unsound cement will cure in air-tight receptacles where no moisture can reach it. This fact points to another cause than embedded CaO for the

condition of unsoundness. In the cement industry the term "overburned clinker" refers to exceedingly hard-burned clinker. From the chemical point of view overburning refers, not to a physical condition but to the chemical condition existing in the clinker when volatilization of essential constituents takes place. Unsoundness in the underburned cement is not due to free CaO, but to $2CaO.SiO_2$, which is very slow setting. In the experiment mentioned, the first set was due to $3CaO.SiO_2$ and $3CaO.Al_2O_3$ which set more quickly than does $2CaO.SiO_2$, then the latter caused the second set. These processes would cause unsoundness when the cement was put to practical uses. The degree of unsoundness is then determined by the per cent of $2CaO.SiO_2$ present, which in turn depends on the temperature of burning. Hence the most effective means of preventing unsoundness is the use of a higher clinkering-tenperature. F. A. KIRKPATRICK.

The cause of unsoundness in cement. RICHARD K. MEADE. *Concrete C. M. S.*, **15**, 70 (1919).—The following are observations based on the practical experience of the writer. Excess of fluxes is of little assistance in producing sound cement. It is not easy to make sound cement from materials high in iron. Since clinker is very porous, the free lime in it should hydrate in the 24-hour storage period of the test pat. Experiment has shown that cement will cure in air-tight receptacles. Neither the free lime theory nor the "dusting clinker" theory are entirely acceptable. Chemists probably do not all mean the same thing when they speak of "the dusting of clinkers." The only "dusting clinker" ever observed by the writer has been produced by a relatively high silica mix. Judging from tests, high lime cements are most apt to be unsound, and they also have the greatest quantity of cohesive constituents. Gypsum, plaster of Paris, or calcium chloride added to cement often make it sound. Assuming that the free lime is locked up inside a minute particle of clinker, do these substances effect its hydration? Concrete may become sound with time; that is, if a number of pats of unsound cement are made at the same time and given the boiling test at intervals of a week, a time is eventually reached when they will stand the test perfectly. A great trouble with all our cement theories is that they explain some things quite well and others not at all. F. A. KIRKPATRICK.

CERAMIC PATENTS.

G. E. MIDDLETON, *Abstractor.*

General.

Kiln. J. C. SCHAFFER. U. S. 1,318,806, October 14, 1919. A series of normally operating, round, down-draft kilns are connected in series by underground and overhead ducts so that they may operate continuously. The products of combustion pass downwardly into the underground duct, thence upwardly through the next adjacent kiln containing green ware, into the overhead duct and out.

Extraction and recovery of potassium or potassium salts from potassium bearing minerals. E. A. Ashcroft. U. S. 1,320,193, October 28, 1919. Potassium or its salts are extracted from minerals such as orthoclase feldspar, micas, alunite, leucite, and from cement-kiln dust, blast-furnace dust, and so forth. The potassium-bearing material mixed with carbon or other reducing agent is treated with chlorin gas in a fused medium as, for example, potassium chloride at a temperature of 800° to 1100° C. The potassium present is thus converted into the normal chloride. A catalyzer, such as ferrous chloride, which absorbs chlorin and passes it on may be used, thus causing greater rapidity of reaction.

Process of recovering potassium salts from silicates. R. S. Edwards. U. S. 1,320,212, October 28, 1919. Potassium-bearing silicates are heated in a rotary kiln with sodium chloride and calcium hydroxide to the fusion point of the chloride. The hydroxide is reduced to the oxide with evolution of H_2O in the presence of which the sodium chloride breaks down and combines with the silicates, forming soluble potassium chloride and with a portion of the silicate as sodium silicate. This mass is discharged into water and the soluble potassium chloride recovered, the calcium oxide being hydrated and thus prepared for further use.

U. S. 1,320,211 discloses substantially the above without the regenerative treatment of the calcium hydroxide. Steam may be used to supply the H_2O necessary for the reaction.

Brick or Tile Press. J. T. and S. Jackson. U. S. 1,320,471, November 4, 1919. A toggle dry press automatically operated to feed, press and discharge. One notable feature is the separate feeding of facing and body material to the mold and the pressing of both in one operation.

Refractories.

Fire-resisting composition. R. F. Bolt. U. S. 1,318,960, October 14, 1919. This composition is designed especially for use in the lower portions of heating furnaces employed in steel mills. It consists of clay, common clay or fire clay, 25% and roll scale 75%.

Refractory material and method of making it. B. Enright. U. S. 1,319,056, October 21, 1919. The objectionable impurity of silica in dolomite used as a basic refractory is eliminated by mixing the dolomite with from 3 to 4% iron ore, pulverizing the mixture and burning it in a rotary kiln at 2700° to 3000° F. The silica is thereby caused to combine with the calcium and magnesium and so rendered innocuous.

Glass.

Means for severing and distributing molten glass. J. E. Potter. U. S. 1,317,176, September 30, 1919. A continuously flowing stream of molten

glass is automatically severed and the successive portions distributed among a series of molds.

The molds A, B, C (see figs.) upon rotary table 3 pass in succession beneath orifice 14 from which molten glass is flowing. Guiding and distributing members 9*a* rotating with the molds deflect the stream of glass and coöperate

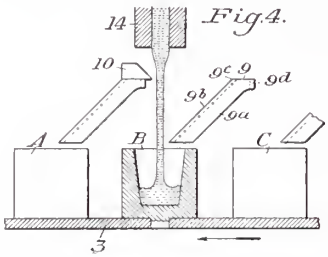

Fig. 4.

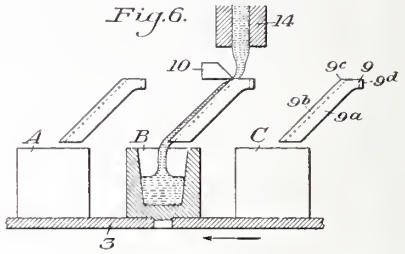

Fig. 6.

at the proper time with fixed member 10 to shear the glass, as indicated in Fig. 6. By this time the succeeding mold is in position to receive its charge and the operation is repeated.

Process of finishing the surfaces of lenses. L. W. Bugbee. U. S. 1,317,481, September 30, 1919. A lens surface is simultaneously polished and treated chemically with the result that the nature of the lens is changed in such a way as to lower its reflection coefficient. A solution of hydrogen sulphide in ammonia mixed with iron peroxide and water is given as an example of the materials used.

High-speed glass-delivery apparatus. A. L. Schram. U. S. 1,317,827, October 7, 1919. The gathering and severing tool is situated immediately below the discharge orifice and the rotary table of the forming machine immediately below this. The forming table bears a series of cups which are passed intermittently beneath the orifice and gathering and severing tool. When these cups are at rest in position to receive a charge, suction means situated beneath the table at that point coöperate with them to accelerate the flow of glass. This construction minimizes the distance of glass travel in the discharge orifice to the mold and thus results in greater speed of operation.

Apparatus and method employed in the manufacture of window-glass cylinders. J. H. Campbell. U. S. 1,319,050, October 7, 1919. In the manufacture of window glass cylinders any variation in temperature of the surface glass in the pot from which the cylinder is being drawn causes the cylinder to move off the center and toward one side of the pot, resulting in an uneven thickness of the cylinder walls. Unsymmetrical heating of the pot before the glass is ladled into it, is frequently the cause of this. In order to obviate this the inventor places the pots upon a rotary table, two of the pots

being under fixed heaters while a third is being drawn from. The heaters are equipped with burners having universally swiveled joints which permit heating of the pots in any desired manner.

Process of making bottles. M. NIVEN. U. S. 1,318,170, October 7, 1919. "Sprinkler bottles," that is, those having neck passages of extremely small bore are made by this process. After the bottle is blown the neck portion is clamped between two jaws and the bottle spun or rotated. The capillary passage is formed by a needle held between the jaws. Lime glass may be used.

Apparatus for manufacturing window-glass cylinders. H. J. WALTER. U. S. 1,318,190, October 7, 1919. A hoisting cage carries a blower having a downwardly projecting air outlet constructed to make an air-tight but yielding and flexible connection with the upper end of the blow pipe carrying the bait. The air pressure required to prevent the collapse of the cylinder is very small, and if during the draw the motor stops the air within the cylinder would be free to escape were it not prevented. By means of this invention the back leaks through the blower and the consequent collapse of the cylinder is prevented.

Glass-forming machine. R. LAFRANCE. U. S. 1,319,098, October 21, 1919. In bottle-blowing machines a head is provided to engage the neck of the bottle and hold it in place during the opening movement of the blow-mold. The head is then withdrawn and the bottle falls without rubbing against and wearing the edge of the mold. Sticking to the mold is also prevented. The head carries a removable disk made of wood which is readily replaceable. As the surface of the wood becomes slightly charred by the hot glass it provides a heat-insulating surface which does not crack the bottle necks.

Apparatus for making sheet-glass. M. J. OWENS. U. S. 1,319,175, October 21, 1919. Molten glass is drawn upwardly in a continuous sheet from the tank and over a bending device to a horizontal position and thence through a lehr. The bending device comprises an endless chain of blocks over which the sheet of glass is drawn. This chain runs continuously in one direction, the blocks being kept moist to prevent overheating by passing through a trough of water on the lower run. This moisture also provides a surface which does not mar the glaze. The edges of the sheets pass over rotary hollow conical drums which prevent catching on the edges of the bending blocks and also prevent sidewise motion of the sheet due to the lateral pull of the moving chain.

Manufacture of sheet glass. J. PROEGER. U. S. 1,320,091, October 28, 1919. In the ordinary processes of drawing sheet glass the outer edges cool more rapidly than the center with resultant irregular shrinkage and warpage. To obviate this the glass is drawn through a heat-distributing casing which utilizes the heat from the furnace in such a way as to insure uniform heat conditions about the sheet and a gradual cooling.

Cylinder-splitting apparatus. C. F. WARRICK. U. S. 1,320,947, November 4, 1919. A burner is reciprocated longitudinally of and beneath the glass cylinder until a cracking tension is created whereupon an abrasive-pointed tool scratches the inside of the cylinder above the heated section and effects the splitting.

Apparatus for transferring glass articles to annealing-furnaces. J. P. MINGLE AND A. G. WILSON. U. S. 1,321,072, November 4, 1919. An apparatus for transferring glass articles such as jars or bottles from an endless conveyor into an annealing lehr. The transferring mechanism comprises a rotary table provided with peripheral semi-cylindrical pockets which receive the jars from the conveyor and deposits them upon the lehr pans.

Glass-mold composition and method of making it. B. C. GILLIGAN. U. S. 1,319,151, October 21, 1919. Molds, plungers, etc., used in molding glassware, are made of a mixture of powdered graphite and retort carbon, bonded with a water solution of caramelized sugar. Molds of this composition are said to produce ware of undimmed luster comparable to that of ware blown without molds.

Faucet for delivering molten glass. C. H. HUBBARD AND G. R. RUPERT. Reissue 14,745, October 28, 1919. Original patent 1,200,195, October 3, 1916.

Lime and Cement.

Gas-fired shaft-kiln. A. AND W. STEIGER. U. S. 1,317,603, September 30, 1919. Lime, magnesite or ores may be calcined in this kiln. The novelty resides primarily in that the cross-section of the shaft is suddenly increased in the cooling zone a considerable amount, preferably immediately below the combustion zone. The air for combustion is introduced near the bottom of the cooling zone and the loosening of the material in this zone due to the larger cross-section facilitates the passage of this air and the cooling of the material.

Rotary kiln for calcining alunite. H. F. CHAPPELL. U. S. 1,317,686, October 7, 1919. This kiln is designed so that the products of combustion of the fuel are completely oxidized before mingling with the oxide of sulphur fumes evolved from the alunite.

Rotary furnace. J. B. GAFFNEY. U. S. 1,318,141, October 7, 1919. Means are provided for removing the material without causing the stopping of the apparatus or the introduction of outside air. That portion of the chamber situated within the furnace is protected from the deterioration caused by combustion gases by a sleeve of refractory material. Means are provided to prevent caking of the material being treated.

Composition matter for building. BRICKS L. HALFER. U. S. 1,317,288, September 30, 1919. This composition comprises sand, crushed stone, screened cinders, granular iron and cement.

Composition for floors. F. ROMANO. U. S. 1,317,525, September 30, 1919. A building material which consists of Portland cement mixed with granite dust or flour.

Plastic composition. B. F. BICKEL. U. S. 1,317,680, October 7, 1919. A composition comprising lime, 9 parts; aluminum sulphate 1 part; a filler; and sufficient liquid to form a plastic mass.

Asbestos cement. H. S. ASHENHURST. U. S. 1,317,853. October 7, 1919. An asbestos cement composed of finely divided particles of the double silicate of magnesium and calcium, having a weight of approximately 25 pounds per cubic foot.

Material for making monolithic structures. B. B. GRUNWALD. U. S. 1,318,075, October 7, 1919. A material consisting of a wet mixture of magnesium oxide, magnesium chloride and a mixture of dead burned and crude, finely-divided magnesite.

Material for closing off water in oil-wells. B. B. GRUNWALD. A material suitable for this purpose consisting of a mixture of a magnesium cement, dead burned and crude finely divided magnesite and a calcium salt.

Method of hydration. W. W. RHODES. U. S. 1,318,921, October 14, 1919. The akaline earth substances to be hydrated are maintained in a moistened condition at a temperature below 100° C, but under pressure such that steam is generated within the mass.

Method of promoting plasticity of mortar materials. W. W. RHODES. U. S. 1,318,922, October 14, 1919. Alkaline hydrates, notably those of calcium and magnesium in pulverulent condition, are subjected to a pressure of only one-half atmosphere for a period of one hour. A denser material is said to result which requires less water to work up into a plaster, and the plaster is said to have greater smoothness and strength and less porosity than one employing untreated hydrates.

Manufacture of portland cement. C. PONTOPPIDAN. U. S. 1,320,172, October 28, 1919. The wet process of manufacturing Portland cement is objectionable in that the slurry often sets to such an extent as to prevent its being handled properly in the apparatus employed. The object of this invention is to make possible the use of the wet process by incorporating a setting preventive with the slurry and subsequently destroying it during calcination. The raw materials such as blast furnace slag and limestone are ground together in water and about a quarter of 1% of sugar added. The sugar acts as a setting preventive but is subsequently eliminated during the clinkering. Molasses and other substances such, for example, as oxy-acids, oxy-aldehydes and oxy-ketones may be used.

NOTE.—Copies of these patents may be obtained for ten cents each by addressing the Commissioner of Patents, Washington, D. C.

AMERICAN CERAMIC SOCIETY.

Acquisition of New Members during November, 1919.

Associate.

Boyden, Mrs. E. S., Librarian, American Optical Co., Southbridge, Mass.

Christopher, Arthur B., Evens & Howard Fire Brick Co., St. Louis, Mo.

Coulston, E. V., Secretary, Rock Island Stove Co., Rock Island, Ill.

Harris, Marshall W., 227 East Park Place, Oklahoma City, Okla.

Hasslacher, George F., Salesman, Roessler & Hasslacher Chem. Co., 400 W. 147th St., New York City.

Heath, Fred T., 4505 Eighteenth Ave., N. E., Seattle, Wash.

Landsheft, C. F., Supt., Vitrolite Co., 939½ Market St., Parkersburg, W. Va.

Sladek, George E., 211 E. Daniels St., Champaign, Ill.

Wey, Harold B., Vice-Pres. and Gen. Mgr., Atlantic Terra Cotta Co., 1302 Third National Bank Bldg., Atlanta, Ga.

Foreign.

Travers, Morris W., D.Sc., F.R.S., Beacon Hall, Priory Garden, Highgate, London, England.

Corporation.

Los Angeles Pressed Brick Co., Frost Bldg., Los Angeles, Cal.

Acquisition of New Members during December, 1919.

Associate.

Beasley, H. C., Gen. Supt., Coonley Mfg. Co., Cicero, Illinois.

Blodgett, Malcolm, Supt., R. Gustavino Co., Woburn, Mass.

Goheen, John P., Sec'y., The Brown Instrument Co., Philadelphia, Pa.

Grace, Richard P., Supt., Mutton Hollow Fire Brick Co., Woodbridge, N. J.

McClafferty, Harry J., 1447 Peach St., Rockford, Ill.

Peck, Albert B., Associate Physicist, Bureau of Standards, Washington, D. C.

Ryan, John F., Sec. and Mgr., Mutton Hollow Fire Brick Co., Woodbridge, N. J.

Ryan, Lawrence C., Asst. Mgr., Mutton Hollow Fire Brick Co., Woodbridge, N. J.

Ryan, Joseph P., Pres., Mutton Hollow Fire Brick Co., Woodbridge, N. J.

Watson, Harold L., Thomas Laboratory, General Electric Co., West Lynn, Mass.

Foreign.

Gentil, E., Engineer, Glaeerie St. Gobain, 1 Bis Place des Saussaics, Paris, France.

Niemura, Nobutaro, Nishimura Chemical Ceramic Laboratory, 1197 Nanba Ashiwarachs, Minamiku. Osaka, Japan.

Corporation.

A. J. Lindemann-Hoverson Co., Milwaukee, Wisconsin.

LOCAL SECTIONS.

Pittsburgh District Section.

The Pittsburgh District Section held its last meeting of the year at the Mellon Institute in Pittsburgh on December 30, 1919. The meeting was called to order at 9.30 A.M. and Mr. Hice presided. Mr. F. H. Riddle had resigned as secretary some time previous to this meeting and Mr. L. R. Office was appointed secretary *pro tem.* Over 35 attended the meeting.

Mr. Bleininger told about the intended visit of members of the English Ceramic Society to the United States next summer and made a motion that the chairman be empowered to appoint a committee, which is to lay out a program for entertaining the English visitors in this district. Motion seconded and passed.

The committee on nominations proposed a list of officers and Mr. J. W. Wright moved to elect the officers as selected by the committee. Motion was seconded and passed unanimously.

Officers for the coming year were elected as follows: Mr. C. R. Peregrine, *Chairman;* Mr. J. Spotts McDowell, *Vice-Chairman;* Mr. Leon R. Office, *Secretary;* Mr. Thos. H. Sant, *Treasurer;* Mr. Francis W. Walker, *Councilor.*

Following the business meeting several very interesting papers were read as follows:

"Note on Glass Refractories," A. V. Bleininger, Bureau of Standards, Pittsburgh, Pa.

"Notes on Clay Pits of England," Thos. H. Sant, John Sant & Sons Co., East Liverpool, Ohio.

"Refractory Cements," Raymond M. Howe, Mellon Institute, Pittsburgh, Pa.

"A Lining for Glass Pots," Dr. S. R. Scholes, H. C. Fry Glass Co., Rochester, Pa.

"Refractories for the By-Product Coke Plant," Leon R. Office, Mellon Institute, Pittsburgh, Pa.

The papers by Messrs. Howe and Office were illustrated by lantern slides

Meeting adjourned.

L. R. OFFICE, *Secretary.*

Chicago Section

At a meeting held in Chicago, on Saturday, November 29th, the Chicago Section of the American Ceramic Society elected the following officers for the ensuing year:

Chairman—Mr. H. T. Bellamy, Western Electric Co., Hawthorne Wks., Chicago, Ill.

Vice-Chairman—Mr. B. S. Radcliffe, Midland Terra Cotta Co., Chicago, Ill.

Chairman Program Committee—Mr. B. T. Sweeley, Western Electric Co., Chicago, Ill.

Chairman Membership Committee—Mr. E. A. Brockman, Roessler and Hasslacher Chem. Co., Chicago, Ill.

Two very interesting papers were given at this meeting, one on "Phosphate Cement," by Prof. R. K. Hursh, of the University of Illinois, and the other on "Notes on Silica Brick," by Mr. Geo. E. Sladek, graduate student of the University of Illinois.

F. L. STEINHOFF, *Secretary-Treasurer.*

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Compiled by Homer F. Staley, C. C. Andrew and A. J. Lambert.

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(D) Discussion
(E) Editorial
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