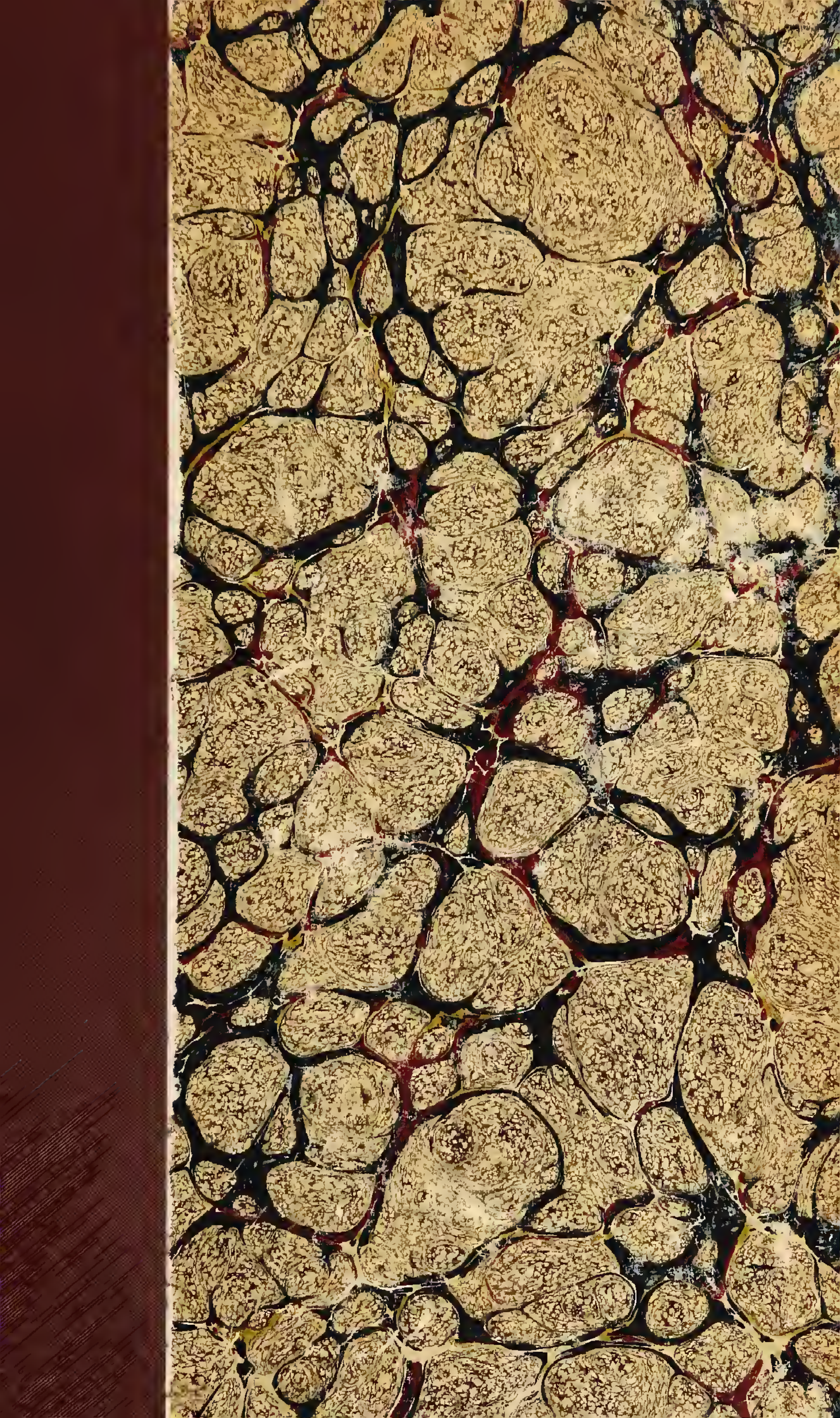

U. S. DEPARTMENT OF AGRICULTURE.

DIVISION OF CHEMISTRY.

BULLETIN No. 13.

FOODS
AND
FOOD ADULTERANTS.

BY DIRECTION OF

THE COMMISSIONER OF AGRICULTURE.

PART FIRST:
DAIRY PRODUCTS.

WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1887.

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LETTER OF SUBMITTAL.

SIR: I have the honor to submit herewith for your inspection and approval Bulletin No. 13, devoted chiefly to a discussion of the best methods of detecting the adulteration of foods.

The first part, which is now placed in your hands, treats of dairy products. Much interest has lately been manifested among those engaged in agriculture in respect of the adulteration of butter, and this part of the subject has been treated with greater detail than any other.

It has been my object in this work to determine the best methods of analysis of the various products in question, and all the recent improvements in analytical methods have been thoroughly tried, and those which have given good results have been adopted in the analytical work which has been done.

Within the last year my division has been supplied with apparatus for photo-micrography, and the illustrations in the following pages are entirely the work of the division unless otherwise stated.

Great benefit has been derived from this method of fixing photographic appearances, as the illustration of the crystalline characters of butters and butter substitutes sufficiently show. The examination of condiments, &c., the report of which will soon follow, was made almost entirely with the microscope, and the illustrations will show how satisfactory this kind of work proves to be. In the matter of photographic illustration no attempt has been made to confine the exhibition to phenomenally fine specimens, but the ordinary appearance of the field of vision has been reproduced. This, I think, is of greater advantage to the general investigation than would be the publication only of the strikingly good negatives. It is believed that by following the methods of analysis recommended in the report it will be possible to detect without fail any adulteration of butter that could possibly prove a commercial success. All other forms of adulteration will be suppressed by the laws of trade. In addition to the report herewith submitted the following parts of the bulletin are almost ready for the press, viz, condiments, sugar, sirup and honey, drinks and canned goods, flour and meal, tea and coffee, and baking powders. Other parts will follow as soon as time is afforded to submit all the process involved to a thorough examination in the laboratory.

Respectfully,

H. W. WILEY,
Chemist.

HON. NORMAN J. COLMAN,
Commissioner of Agriculture.

CONTENTS.

BUTTER AND ITS ADULTERATIONS.

The adulteration of butter with other fats has of late years attracted the attention not only of the analyst but also of the political economist and health officer.

This matter has been deemed of sufficient importance to demand regulation by law of Congress. This law provides for the inspection and analysis of commercial butters and their substitutes.

Following is the text of the act:

An Act defining butter, also imposing a tax upon and regulating the manufacture, sale, importation, and exportation of oleomargarine.

Be it enacted by the Senate and House of Representatives of the United States of America in Congress assembled, That for the purposes of this act the word "butter" shall be understood to mean the food product usually known as butter, and which is made exclusively from milk or cream, or both, with or without common salt, and with or without additional coloring matter.

Sec. 2. That for the purposes of this act certain manufactured substances, certain extracts, and certain mixtures and compounds, including such mixtures and compounds with butter, shall be known and designated as "oleomargarine," namely: All substances heretofore known as oleomargarine, oleo, oleomargarine-oil, butterine, lardine, suine, and neutral; all mixtures and compounds of oleomargarine, oleo, oleomargarine-oil, butterine, lardine, suine, and neutral; all lard extracts and tallow extracts; and all mixtures and compounds of tallow, beef-fat, suet, lard, lard-oil, vegetable-oil, annotto, and other coloring matter, intestinal fat, and offal fat made in imitation or semblance of butter, or when so made, calculated, or intended to be sold as butter or for butter.

Sec. 3. That special taxes are imposed as follows:

Manufacturers of oleomargarine shall pay six hundred dollars. Every person who manufactures oleomargarine for sale shall be deemed a manufacturer of oleomargarine.

Wholesale dealers in oleomargarine shall pay four hundred and eighty dollars. Every person who sells or offers for sale oleomargarine in the original manufacturer's packages shall be deemed a wholesale dealer in oleomargarine. But any manufacturer of oleomargarine who has given the required bond and paid the required special tax, and who sells only oleomargarine of his own production, at the place of manufacture, in the original packages to which the tax-paid stamps are affixed, shall not be required to pay the special tax of a wholesale dealer in oleomargarine on account of such sales.

Retail dealers in oleomargarine shall pay forty-eight dollars. Every person who sells oleomargarine in less quantities than ten pounds at one time shall be regarded as a retail dealer in oleomargarine; and sections thirty-two hundred and thirty-two, thirty-two hundred and thirty-three, thirty-two hundred and thirty-four, thirty-two hundred and thirty-five, thirty-two hundred and thirty-six, thirty-two hundred and thirty-seven, thirty-two hundred and thirty-eight, thirty-two hundred and thirty-nine, thirty-two hundred and forty, thirty-two hundred and forty-one, and thirty-two hundred and forty-three of the Revised Statutes of the United States are, so far as applicable, made to extend to and include and apply to the special taxes imposed by this section, and to the persons upon whom they are imposed: *Provided*, That in case any manufacturer of oleomargarine commences business subsequent to the thirtieth day of June in any year, the special tax shall be reckoned from the first day of July in that year, and shall be five hundred dollars.

SEC. 4. That every person who carries on the business of a manufacturer of oleomargarine without having paid the special tax therefor, as required by law, shall, besides being liable to the payment of the tax, be fined not less than one thousand and not more than five thousand dollars; and every person who carries on the business of a wholesale dealer in oleomargarine without having paid the special tax therefor, as required by law, shall, besides being liable to the payment of the tax, be fined not less than five hundred nor more than two thousand dollars; and every person who carries on the business of a retail dealer in oleomargarine without having paid the special tax therefor, as required by law, shall, besides being liable to the payment of the tax, be fined not less than fifty nor more than five hundred dollars for each and every offense.

SEC. 5. That every manufacturer of oleomargarine shall file with the collector of internal revenue of the district in which his manufactory is located such notices, inventories, and bonds, shall keep such books and render such returns of material and products, shall put up such signs and affix such number to his factory, and conduct his business under such surveillance of officers and agents as the Commissioner of Internal Revenue, with the approval of the Secretary of the Treasury, may, by regulation, require. But the bond required of such manufacturer shall be with sureties satisfactory to the collector of internal revenue, and in a penal sum of not less than five thousand dollars; and the sum of said bond may be increased from time to time, and additional sureties required at the discretion of the collector, or under instructions of the Commissioner of Internal Revenue.

SEC. 6. That all oleomargarine shall be packed by the manufacturer thereof in firkins, tubs, or other wooden packages not before used for that purpose, each containing not less than ten pounds, and marked, stamped, and branded as the Commissioner of Internal Revenue, with the approval of the Secretary of the Treasury, shall prescribe; and all sales made by the manufacturers of oleomargarine, and wholesale dealers in oleomargarine, shall be in original stamped packages. Retail dealers in oleomargarine must sell only from original stamped packages, in quantities not exceeding ten pounds, and shall pack the oleomargarine sold by them in suitable wooden or paper packages, which shall be marked and branded as the Commissioner of Internal Revenue, with the approval of the Secretary of the Treasury, shall prescribe. Every person who knowingly sells or offers for sale, or delivers or offers to deliver, any oleomargarine in any other form than in new wooden or paper packages as above described, or who packs in any package any oleomargarine in any manner contrary to law, or who falsely brands any package or affixes a stamp on any package denoting a less amount of tax than that required by law, shall be fined for each offense not more than one thousand dollars, and be imprisoned not more than two years.

SEC. 7. That every manufacturer of oleomargarine shall securely affix, by pasting, on each package containing oleomargarine manufactured by him, a label on which shall be printed, besides the number of the manufactory and the district and State in which it is situated, these words: "Notice—The manufacturer of the oleomargarine herein contained has complied with all the requirements of law. Every person is cautioned not to use either this package or the stamp thereon again, nor to remove the contents of this package without destroying said stamp, under the penalty provided by law in such cases." Every manufacturer of oleomargarine who neglects to affix such label to any package containing oleomargarine made by him, or sold or offered for sale by or for him, and every person who removes any such label so affixed from any such package, shall be fined fifty dollars for each package in respect to which such offense is committed.

SEC. 8. That upon oleomargarine which shall be manufactured and sold, or removed for consumption or use, there shall be assessed and collected a tax of two cents per pound, to be paid by the manufacturer thereof; and any fractional part of a pound in a package shall be taxed as a pound. The tax levied by this section shall be represented by coupon stamps; and the provisions of existing laws governing the engraving, issue, sale, accountability, effacement, and destruction of stamps relating to tobacco and snuff, as far as applicable, are hereby made to apply to stamps provided for by this section.

SEC. 9. That whenever any manufacturer of oleomargarine sells, or removes for sale or consumption, any oleomargarine upon which the tax is required to be paid by stamps, without the use of the proper stamps, it shall be the duty of the Commissioner of Internal Revenue, within a period of not more than two years after such sale or removal, upon satisfactory proof, to estimate the amount of tax which has been omitted to be paid, and to make an assessment therefor and certify the same to the collector. The tax so assessed shall be in addition to the penalties imposed by law for such sale or removal.

SEC. 10. That all oleomargarine imported from foreign countries shall, in addition to any import duty imposed on the same, pay an internal-revenue tax of fifteen cents per pound, such tax to be represented by coupon stamps as in the case of oleomargarine manufactured in the United States. The stamps shall be affixed and canceled by the owner or importer of the oleomargarine while it is in the custody of the proper custom-house officers; and the oleomargarine shall not pass out of the custody of said officers until the stamps have been so affixed and canceled, but shall be put up in wooden packages, each containing not less than ten pounds, as prescribed in this act for oleomargarine manufactured in the United States, before the stamps are affixed; and the owner or importer of such oleomargarine shall be liable to all the penal provisions of this act prescribed for manufacturers of oleomargarine manufactured in the United States. Whenever it is necessary to take any oleomargarine so imported to any place other than the public stores of the United States for the purpose of affixing and canceling such stamps, the collector of customs of the port where such oleomargarine is entered shall designate a bonded warehouse to which it shall be taken, under the control of such customs officer as such collector may direct; and every officer of customs who permits any such oleomargarine to pass out of his custody or control without compliance by the owner or importer thereof with the provisions of this section relating thereto, shall be guilty of a misdemeanor, and shall be fined not less than one thousand dollars nor more than five thousand dollars, and imprisoned not less than six months nor more than three years. Every person who sells or offers for sale any imported oleomargarine, or oleomargarine purporting or claimed to have been imported, not put up in packages and stamped as provided by this act, shall be fined not less than five hundred dollars nor more than five thousand dollars, and be imprisoned not less than six months nor more than two years.

SEC. 11. That every person who knowingly purchases or receives for sale any oleomargarine which has not been branded or stamped according to law shall be liable to a penalty of fifty dollars for each such offense.

SEC. 12. That every person who knowingly purchases or receives for sale any oleomargarine from any manufacturer who has not paid the special tax shall be liable for each offense to a penalty of one hundred dollars, and to a forfeiture of all articles so purchased or received, or of the full value thereof.

SEC. 13. That whenever any stamped package containing oleomargarine is emptied, it shall be the duty of the person in whose hands the same is to destroy utterly the stamps thereon; and any person who willfully neglects or refuses so to do shall for each such offense be fined not exceeding fifty dollars, and imprisoned not less than ten days nor more than six months. And any person who fraudulently gives away or accepts from another, or who sells, buys, or uses for packing oleomargarine, any such stamped package, shall for each such offense be fined not exceeding one hundred dollars, and be imprisoned not more than one year. Any revenue officer may destroy any emptied oleomargarine package upon which the tax-paid stamp is found.

SEC. 14. That there shall be in the office of the Commissioner of Internal Revenue an analytical chemist and a microscopist, who shall each be appointed by the Secretary of the Treasury, and shall each receive a salary of two thousand five hundred dollars per annum; and the Commissioner of Internal Revenue may, whenever in his judgment the necessities of the service so require, employ chemists and microscopists, to be paid such compensation as he may deem proper, not exceeding in the aggregate any appropriation made for that purpose. And such Commissioner is authorized to decide what substances, extracts, mixtures, or compounds which may be submitted for his inspection in contested cases are to be taxed under this act; and his decision in matters of taxation under this act shall be final. The Commissioner may also decide whether any substance made in imitation or semblance of butter, and intended for human consumption, contains ingredients deleterious to the public health; but in case of doubt or contest his decisions in this class of cases may be appealed from to a board hereby constituted for the purpose, and composed of the Surgeon-General of the Army, the Surgeon-General of the Navy, and the Commissioner of Agriculture; and the decisions of this board shall be final in the premises.

SEC. 15. That all packages of oleomargarine subject to tax under this act that shall be found without stamps or marks as herein provided, and all oleomargarine intended for human consumption which contains ingredients adjudged, as hereinbefore provided, to be deleterious to the public health, shall be forfeited to the United States. Any person who shall willfully remove or deface the stamps, marks, or brands on package containing oleomargarine taxed as provided herein shall be guilty of a misdemeanor, and shall be punished by a fine of not less than one hundred dollars nor more than two thousand dollars, and by imprisonment for not less than thirty days nor more than six months.

SEC. 16. That oleomargarine may be removed from the place of manufacture for export to a foreign country without payment of tax or affixing stamps thereto, under such regulations and the filing of such bonds and other security as the Commissioner of Internal Revenue, with the approval of the Secretary of the Treasury, may prescribe. Every person who shall export oleomargarine shall brand upon every tub, firkin, or other package containing such article the word "oleomargarine," in plain Roman letters not less than one half inch square.

SEC. 17. That whenever any person engaged in carrying on the business of manufacturing oleomargarine defrauds, or attempts to defraud, the United States of the tax on the oleomargarine produced by him, or any part thereof, he shall forfeit the factory and manufacturing apparatus used by him, and all oleomargarine and all raw material for the production of oleomargarine found in the factory and on the factory premises, and shall be fined not less than five hundred dollars nor more than five thousand dollars, and be imprisoned not less than six months nor more than three years.

SEC. 18. That if any manufacturer of oleomargarine, any dealer therein or any importer or exporter thereof shall knowingly or willfully omit, neglect, or refuse to do, or cause to be done, any of the things required by law in the carrying on or conducting of his business, or shall do anything by this act prohibited, if there be no specific penalty or punishment imposed by any other section of this act for the neglecting, omitting, or refusing to do, or for the doing or causing to be done, the thing required or prohibited, he shall pay a penalty of one thousand dollars; and if the person so offending be the manufacturer of or a wholesale dealer in oleomargarine, all the oleomargarine owned by him, or in which he has any interest as owner, shall be forfeited to the United States.

SEC. 19. That all fines, penalties, and forfeitures imposed by this act may be recovered in any court of competent jurisdiction.

SEC. 20. That the Commissioner of Internal Revenue, with the approval of the Secretary of the Treasury, may make all needful regulations for the carrying into effect of this act.

SEC. 21. That this act shall go into effect on the ninetieth day after its passage; and all wooden packages containing ten or more pounds of oleomargarine found on the premises of any dealer on or after the ninetieth day succeeding the date of the passage of this act shall be deemed to be taxable under section eight of this act, and shall be taxed, and shall have affixed thereto the stamps, marks, and brands required by this act or by regulations made pursuant to this act; and for the purposes of securing the affixing of the stamps, marks, and brands required by this act, the oleomargarine shall be regarded as having been manufactured and sold, or removed from the manufactory for consumption or use, on or after the day this act takes effect; and such stock on hand at the time of the taking effect of this act may be stamped, marked, and branded under special regulations of the Commissioner of Internal Revenue, approved by the Secretary of the Treasury; and the Commissioner of Internal Revenue may authorize the holder of such packages to mark and brand the same and to affix thereto the proper tax-paid stamps.

Approved, August 2, 1886.

ARTIFICIAL BUTTER.

The French chemist, Mége-Mouries, in 1870 first described a method of making artificial butter on a large scale.

Mége, who was employed on the Imperial farm at Vincennes, was led to undertake this study through a desire to furnish to the poorer classes and to sailors an article which should be cheaper and more stable in its composition than ordinary butter.

He endeavored to imitate the physiological process which he supposed took place when cows were insufficiently fed, and when, therefore, the butter which they furnished was derived from their own fat. From beef he obtained a fat "which melted at almost the exact temperature of butter, possessed a sweet and agreeable taste, and which for most purposes could replace ordinary butter, not, of course, the finest kinds, but which was superior to it in possessing the advantageous peculiarity of keeping for a long time without becoming rancid."

Before the breaking out of the Franco-Prussian war Mége had established a factory at Poissy. The war suspended the operations of this factory, but at the cessation of hostilities they were again commenced.

Following is the method employed in the year 1873, in the manufacture of artificial butters:

The fat of best quality from recently killed bullocks is finely cut in a kind of sausage grinder in order to break up the membranes. The fragments fall into a tank heated with steam, which for every 1,000 parts of fat contains 300 parts of water and 1 part of carbonate of potash and 2 stomachs of sheep or pigs.

The temperature of the mixture is raised to 45° C. After two hours, under the influence of the pepsin in the stomachs, the membranes are dissolved and the fat melted and risen to the top of the mixture.

The fat is next drawn off into a second tank, kept at a somewhat higher temperature, and 2 per cent. of common salt added. After two hours more the fat becomes clear and takes on a yellow color and acquires somewhat the taste and odor of fresh butter. The fat is now drawn off into vessels and allowed to cool. It is then cut into pieces, wrapped in linen, and put in a hydraulic press and kept at a temperature of about 25° C. By pressure the fat is separated into two portions, viz: stearine 40 to 50 per cent., and fluid oleo 50 to 60 per cent. The stearine remaining in the presses is used in candle-making. Mége's patent, possessing as it does historical interest, is given in full.

A full citation of the various patents taken out in foreign countries is found in "Sell's Kunstbutter."[1]

The patents taken out in this country for the manufacture of artificial butter are given below:

LIST OF PATENTS GRANTED IN THE UNITED STATES FOR THE MANUFACTURE OF BUTTER SUBSTITUTES.

Hippolyte Mége, No. 146012, *dated December* 30, 1873.

To all whom it may concern:

Be it known that I, Hippolyte Mége, of Paris, France, have discovered a new and improved process of transforming animal fats into butter, of which the following is a full, clear, and exact description:

The butter which is obtained from milk is produced by the cow elaborating her own fat through her cellular mammary tissues at the low rate of temperature of the body.

The animal fat from which the butter-cells in milk are produced is composed chiefly of oleine, margarine, and stearine, and small quantities of other substances.

The natural process performed by the cow consists, mainly, first, in separating the oleomargarine from the stearine without developing disagreeable odors or flavors in the oleomargarine; and, secondly, in producing a slight change in the oleomargarine, by which it assumes the character of butter.

My invention, hereinafter described, is based upon a discovery made by me, that when the fat is rendered at a low temperature, considerably below that heretofore employed in the ordinary rendering of fat, it has the taste of molten butter, and does not acquire that peculiarly disagreeable flavor heretofore supposed to be necessarily attached to melted fat or tallow, and which is designated as "tallowy flavor."

I have succeeded in obtaining excellent results by rendering the crude fat at a temperature of 103° Fahrenheit, which is below the temperature at which the tallowy flavor is created. The temperature may be raised above this point in order to facilitate the operation, provided care be taken to avoid attaining the temperature at which the tallowy flavor is created.

The precise limit to which it is safe to increase the rendering-temperature can be ascertained by trial under various circumstances with the different kinds of fat. The temperature must, however, be far below that heretofore ordinarily used in rendering fats when no such object as I propose—to wit, the making of a butter-like product—was had in view. I do not think it would be safe to vary many degrees above that specifically indicated.

I have also discovered that, in order to neutralize any fermentation of the fat before or during its treatment, the raw fat should, as soon as possible after the death of the animal, be plunged in a solution of fifteen (15) per cent. of common salt and one per cent. of sulphate of soda, the effect of which would be to prevent such fermentation.

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[1] Arbeiten a. d. Kaiserlichen Gesundheitsamte, pp. 481–493.

In carrying out my process I first crush, grind, or disintegrate the fat by any suitable machinery, such as rollers or millstones, in order to break up the cellular tissues in which the fat is contained in the animal, and thus cause it to be more easily melted or rendered by the application of low temperatures. This fat thus disintegrated is to be slowly raised to a temperature of 103° Fahrenheit in a vessel in which the temperature can be raised at will until the rendering shall be complete. The temperature, as before stated, must be so regulated that the rendered fat will have the taste of molten butter, and care should be taken not to heat it so as to induce the change which produces the usual disagreeable taste of melted fat or tallow, instead of the taste of molten butter, which temperature is considerably below that heretofore ordinarily used in rendering fat, and will be found to vary not many degrees above the point already stated.

I also add to the fat while being rendered, for the purpose of aiding in this process, two liters of gastric juice to a hundred (100) kilograms of fat. This gastric juice is made by macerating, for three hours, half of the stomach of a pig or sheep, well washed, and three litres of water containing thirty grams of bi-phosphate of lime. After maceration this macerated substance is passed through a sieve, and then added to the fat under treatment in the proportion of two litres to one hundred (100) kilograms.

The separation of the organized tissues from the fat is aided by the introduction of salt during the rendering; and as soon as there are no lumps of fat visible in the kettle I add about one per cent. of common salt. I stir it for some time. The rendered fat is then allowed to stand until it attains perfect limpidity, when it can be drawn off. By this means the separation is well made, and the organized tissues which do deposit are not altered. I then allow the melted fat to stand in a vessel, maintained at a temperature of about 86° to 98°, until the stearine is crystallized. The mixture of stearine and oleomargarine may then be put in a centrifugal machine; and by the operation of this machine the oleomargarine will pass through the cloth and the stearine remain within; or the mixture may be subjected to pressure in a press. The effect produced in either case is that the oleomargarine practically separates from the stearine and flows out. The oleomargarine thus separated from the stearine, when cooled, constitutes a fatty matter of very good taste, which may replace the butter used in the kitchen. If it is desired, however, to transform it into more perfect butter, I employ the following means: I mix the oleomargarine, as it comes from the press or centrifugal machine, with milk and cream, equal to ten per cent. of the weight of the oleomargarine, the temperature of the milk and cream being about seventy-one (71) degrees, and thoroughly agitate them together. I then let the mixture become completely cold and solid, and then cause it to be worked between rollers, which give it the homogeneousness and the consistency which are the qualities of the natural butter.

The above process of agitating the oleomargarine with milk is intended to be adopted when the butter is to be immediately used. If the butter is intended to be preserved, it will be better to mix the oleomargarine at animal heat with ten per cent. of its weight of water instead of milk or cream, and then agitate the two together, as above described.

I have also found it expedient to mix with the cream or milk, in the first case above described, before agitating, or with the water in the other case above described, before agitating, a fiftieth part of mammary tissue, which is the udder of the cow, minced fine, a one-hundredth part of bicarbonate of soda, and some coloring matter.

It may be desirable to add ordinary butter, and this I do by mixing the oleomargarine and the ordinary butter together at a temperature of about 70° Fahrenheit.

What I claim as my invention, and desire to secure by Letters Patent, is—

(1) The rendering of animal fat at a low temperature, substantially as above set forth, for the production of a fatty matter devoid of disagreeable taste.

(2) As a new product of manufacture, fat rendered at the low temperature, substantially as above described, devoid of disagreeable taste.

(3) The combined process of rendering animal fat at a low temperature and then separating the oleomargarine for the purpose of producing a material adapted to be used as ordinary butter for culinary purposes, or to be further treated for making more perfect butter, substantially as above described.

(4) As a new product of manufacture, oleomargarine obtained from fat rendered at a low temperature and separated from the stearine, substantially as above described.

(5) The agitating of oleomargarine with water or milk for the purpose of making a more perfect imitation of butter, substantially as above described.

(6) The butter-like product produced by the agitation of oleomargarine with water or milk, substantially as above described.

(7) The treatment with artificial gastric juice for facilitating the process of rendering the fat at a low temperature, substantially as above described.

(8) The treatment of the oleomargarine with the mammary tissue of the cow, or mammary pepsin, substantially as above described.

(9) The addition of ordinary butter to oleomargarine, substantially as above described.

H. MÉGE.

Witnesses:
ROBT. M. HOOPER,
M. D. DESHLER.

William E. Andrew, No. 153,999, *dated August* 11, 1874.

The process, herein described, for rendering fats, consisting in the application of dry heat or dry hot air to liquefy, and pressure to separate, the oily portion from the membrane, and removing the liquid portions from contact with the membranous portions as fast as separated.

William E. Andrew, No. 166,955, *dated August* 24, 1875.

Complete process of manufacturing artificial butter, herein described, consisting first in rupturing and destroying the globular condition of animal oil by agitation and then refrigerating the same, then combining the product thus obtained with butter, cream, or milk and churning until a thorough amalgamation takes place.

William E. Andrew, No. 172,942, *dated February* 1, 1876.

The process of clarifying liquid tallow or oil by injecting into the oil, under force, in the form of mist or fine spray, water prepared with chloride of sodium or nitrate of potash, and heated to a higher degree of temperature than the oil.

Garret Cosine, No. 173,591, *dated February* 15, 1876.

The process of making artificial butter by mixing together oleine and margarine from animal fats, and from fruit and vegetable nuts, and lactic acid and loppered cream or milk.

William E. Andrew, No. 179,883, *dated July* 18, 1876.

(Mechanical.)

Alfred Springer, No. 187,327, *dated February* 13, 1877.

The process of producing edible fat or tallow by heating the crude fat at a temperature of 140° to 145° Fahrenheit, in contact with common salt, saltpeter, borax, and boracic and salicylic acids, withdrawing the separated fat and incorporating therewith a second and smaller charge of the above chemicals, with the addition of benzoic acid.

Amor Smith, No. 188,428, *dated March* 13, 1877.

Method of separating oleomargarine from the fat of kine, that is to say, by separating it directly from the stearine and membrane at a low heat, without having first heated the mass to a higher point, for the purpose of removing the membrane from the stearine and oleine.

Royal W. Barnard, No. 198,334, *dated December* 18, 1877.

Method of reclaiming sour "tubby," or rancid butter, which consists in treating the same with a solution of brine containing an alkaline carbonate mixed with a solution of tartaric acid, or its equivalent.

Thomas F. Wilkins, No. 226,467, *dated April* 13, 1880.

Butter containing metaphosphoric acid intimately incorporated therewith, whereby the butter is preserved.

Otto Roysen, No. 236,483, *dated January* 11, 1881.

Process of making a substitute for butter, consisting in adding to oleomargarine an alkaline solution, and agitating the mixture until partial saponification ensues, and then adding a minute quantity of butyric acid.

Thomas F. Wilkins, No. 9,892, *reissued, dated October* 11, 1881.

The method herein described of preserving fats and other organic matter by mechanically mingling phosphoric acid therewith.

Samuel H. Cochran, No. 258,992, *dated June* 6, 1882.

The combination ot beef-suet oil, cotton-seed oil and its equivalents, purified and flavored as described, with beef-stearine and slippery-elm bark.

Hippolyte Mége, No. 10,137, *reissued, dated June* 13, 1882.

Treating animal fats so as to remove the tissues and other portions named, with or without the addition of substances to change the flavor.

Samuel H. Cochran, No. 10,171, *reissued, dated August* 1, 1882.

A combination of beef-suet oil, cotton-seed oil and its equivalents, with beef-stearine.

Samuel H. Cochran, No. 262,207, *dated August* 8, 1882.

Compound composed of the oil obtained from swine fat, cotton-seed oil and its equivalents, deodorized and purified by slippery-elm bark and beef-stearine.

John Hobbs, No. 263,042, *dated August* 22, 1882.

The vegetable stearine to be used can be obtained from any pure vegetable, seed, or nut oils by pressing them at a temperature as above set forth, or it may be obtained in the market at times as vegetable stearine.

Mixing "vegetable stearine" or "margarine" obtained substantially as described, with what is called "animal oleomargarine" and emulsionizing the said mixture with milk, cream or other watery fluid.

Nathan I. Nathan, No. 263,199, *dated August* 22, 1882.

Process of manufacturing artificial butter by uniting oleomargarine with leaf lard, the latter having been previously cleaned, fused, strained, and subjected to a washing action in a solution of water, borax, and nitric acid, then rewashed and the united mass heated and subjected to the ordinary churning operation.

George S. Marshall, No. 264,545, *dated September* 19, 1882.

Process of deodorizing, purifying and flavoring stearine obtained from animal fats, or vegetable oils, by boiling the same with water and mixing therewith powdered orris-root.

William Cooley, No. 264,516, *dated September* 19, 1882.

An artificial cream composed of an oleaginous substance mechanically blended, or otherwise incorporated with milk, buttermilk, or cream, the oleaginous material being in a state of minute and even division, and each particle encased in a coating of caseine.

Henry Lauferty, No. 265,833, *dated October* 10, 1882.

Improvement in the manufacture of artificial butter, or oleomargarine, which consists in treating in the manner described both the milk and the oleomargarine oil separately with sal-soda, prepared and taken in the proportions as specified, then mixing or churning the creamy substance produced from the treated milk with the prepared oleomargarine oil, and coloring, salting, and working the mixture.

Hugo Berthold, No. 266,417, *dated October* 24, 1882.

A coloring compound for admixture with oleomargarine oil after the usual churning operation, consisting of saccharine matter, glycerine, annotto, and oil of ben, mixed togther.

George H. Webster, No. 266,568, *dated October* 24, 1882.

Process of making artificial butter, which consists in minutely dividing leaf-lard, rendering and straining it, mixing a butter-coloring matter with it, immersing it for thirty-six hours in cold brine, transferring it from the brine to dry tables or shelves and keeping it there covered with salt for thirty-six hours; then heating it to about 130° Fahrenheit and mixing it with lukewarm buttermilk, a small quantity of clarified tallow, and a minute quantity of pepsin, and allowing the mixture to settle; then transferring the liquid lard and tallow to a vessel containing comminuted butter of about half the weight of the lard, thoroughly mixing the contents of the vessel by stirring, pouring the mixture into cold water, and thoroughly working it in the usual manner.

William H. Burnett, No. 266,580, *dated October* 24, 1882.

The butter-like product described, consisting of the ingredients specified, to wit, lard, beef-suet, butter, glycerine, salt water, and coloring material.

Oscar H. Coumbe, No. 266,778, *dated October* 31, 1882.

A new article of manufacture, oleard, consisting of vegetable oil, in combination with cooked farinaceous flour.

Oscar H. Coumbe, No. 266,777, *dated October* 31, 1882.

An improved article of commerce known as butteroid, and consisting of cottonseed or other vegetable oil treated with a solution of caustic soda, in combination with farinaceous flour first thoroughly cooked in salt water.

Henry R. Wright, No. 267,637, *dated November* 14, 1882.

Process of making artificial butter or creamine, which consists in mixing together the oils derived from animal fat at low temperatures with sweet cream, the oil of butter, vegetable oil, and coloring matter; then allowing these ingredients to become sour while together; then removing the whey, and finally churning the mass.

Joseph H. McDonald, No. 270,454, *dated January* 9, 1883.

(Mechanical.)

John Hobbs, No. 271,239, *dated January* 30, 1883.

(Mechanical.)

John Hobbs, No. 271,240, *dated January* 30, 1883.

(Mechanical.)

John Hobbs, No. 271,244, *dated January* 30, 1883.

(Mechanical.)

John Hobbs, No. 271,241, *dated January* 30, 1883.

(Mechanical.)

John Hobbs, No. 271,243, *dated January* 30, 1883.

(Mechanical.)

John Hobbs, No. 271,242, *dated January* 30, 1883.

(Mechanical.)

John Hobbs, No. 280,822, *dated July* 10, 1883.

Process of refining fats, which consists in first finely grinding the fat of the leaf of the hog, mixing it thoroughly with salt, placing it in tanks of cold water for two or three days, when it is worked over, as described, then rendering it at a low temperature, and as quickly as possible, with or without adding the solution mentioned, then drawing it off from the tissue, clarifying it and again drawing it off and cooling it.

Samuel H. Cochran, No. 285,878, *dated October* 2, 1883.

The mode above described of giving a butter-flavor to animal fats or oils, which consists in mixing therewith in the manner above described a quantity of dairy or creamery butter in its normal or hard condition.

Samuel H. Cochran, No. 285,973, *dated October* 2, 1883.

(Mechanical.)

Samuel H. Cochran, No. 285,974, *dated October* 2, 1883.

(Mechanical.)

Andrew J. Chase, No. 286,778, *dated October* 16, 1883.

The method herein described of manufacturing butter from animal oils, said method consisting in subjecting the oils to a low temperature, and at the same time agitating them, both during the process of solidifying and afterwards.

John Hobbs, No. 289,100, *dated November* 27, 1883.

The manufacture of deodorized fats or oxyline, the use or employment of the substance herein mentioned—vegetable stearine—in combination with the other ingredients named--oleomargarine-stearine and oleomargarine-stock.

George Lawrence, No. 295,180, *dated March* 18, 1884.

Process of treating milk with fatty and other matters by passing it and them, mingled with gases, through one or more steam-ejectors, for separating and mixing the particles.

Samuel Schwarzschild, No. 299,685, *dated June* 3, 1884.

(Mechanical.)

Emma J. Woodruff, No. 327,636, *dated October* 6, 1885.

Adding to the milk white-wine rennet, sugar, salt, bicarbonate of soda, bicarbonate potassium, alum, and butter.

Lyman Guinnip, No. 334,430, *dated January* 19, 1886.

Consisting in mingling two bodies of cream of different age, then churning the same, then removing a portion thereof from the churn and mingling with the removed part a quantity of butter, then churning the residue until butter begins to separate, then adding butter thereto, as specified, and churning the mixture, and finally adding thereto the portion first abstracted, and churning the whole until the butter is made.

William A. Murray, No. 335,084, *dated January* 26, 1886.

Mixing 1 gallon of sweet milk with 1 ounce of liquid rennet, 25 grains (Troy) of nitrate of potash, 1 ounce granulated sugar, half-teaspoonful of butter-coloring, and 8 pounds of butter, churned together and worked.

Carl August Johansson, No. 336,324, *dated February* 16, 1886.

(Mechanical.)

George Wm. Sample, No. 336,438, *dated February* 16, 1886.

(Mechanical.)

Charles Marchand, No. 338,538, *dated March* 23, 1886.

(Mechanical.)

Edward J. Oatman, No. 346,062, *dated July* 20, 1886.

Producing an emulsion from milk or its derivates and a suitable oleaginous material, which consists in thoroughly dividing and commingling the ingredients by injecting a steam jet into the mixture, The common method of manufacture employed in this country is set forth by Armsby:[1]

> Although numerous patents have been taken out for the manufacture of imitation butter, and a great variety of materials have been named in the specifications, the process as now conducted is comparatively simple. The raw materials are beef-tallow, leaf-lard, and the best quality of butter, together with small amounts of milk or cream and of butter-color.
>
> From the beef-tallow is prepared the oleomargarine oil of Mége. The caul fat of freshly killed beeves is, after thorough washing first in tepid and then in iced water, allowed to stand in a cold room until thoroughly cold. It is then rendered at a temperature between 130° and 175° F. The resulting oil is allowed to cool slowly until a considerable portion of the stearine and palmitin have crystallized out, and the pasty mass is then subjected to hydraulic pressure. The still fluid (about two-thirds of the whole) flows out into a tank of cold water, where it solidifies into a granular mass which is known in the trade as "oleo" oil or simply "oleo". The name "oil" is somewhat misleading, as the product is a granular solid of a slightly yellow color. Fresh leaf-lard treated in substantially the same way as the beef-tallow, yields the "neutral lard" or "neutral" of the trade, also a granular solid of a white color.
>
> The objects of this treatment are twofold: first, to produce fats as free as possible from taste or odor; second, to remove some of the difficultly fusible stearine and palmitin, in order that the finished product may melt readily in the mouth.
>
> Having thus secured the fats in proper condition, the manufacturer proceeds to mix the "oleo" and "neutral", the proportions varying according to the destination of product; a warm climate calling for more "oleo," a cold one for more "neutral," and to flavor the mixture with butter. This flavoring is conducted in large, steam-jacketed vessels provided with revolving paddles, by which their contents can be thoroughly agitated. Here the "oleo" and "neutral" are melted and thoroughly agitated with a certain proportion of milk, or sometimes of cream, and a proper amount of butter-color. Forty-eight gallons of milk per 2,000 pounds of product are stated to be a common proportion. After sufficient agitation, the melted mass is run into cold water, and as it cools is broken up by paddles so as to granulate the mass. After thorough washing, it is salted and worked exactly like butter. The product is known as oleomargarine. Although it contains hardly more than a trace of butter fat, the latter flavors the whole mass so strongly that, when well salted, as it usually is, it might readily pass with an inexpert or careless consumer for a rather flavorless butter. Oleomargarine is the cheapest product made. By adding to the material in the agitator or "churn" more or less pure butter, what is known as butterine is produced, two grades of which are commonly sold, viz, "creamery butterine" containing more, and "dairy butterine" containing less butter.

The method of manufacture used by the firm of Armour & Co., of Chicago, is thus described by Mr. Philip D. Armour:[2]

> The fat is taken from the cattle in the process of slaughtering, and after thorough washing is placed in a bath of clean, cold water, and surrounded with ice, where it is allowed to remain until all animal heat has been removed. It is then cut into small pieces by machinery and cooked at a temperature of about 150° until the fat, in liquid form, has separated from the fibrine or tissue, then settled until it is perfectly clear. Then it is drawn into graining vats and allowed to stand a day, when it is ready for the presses. The pressing extracts the stearine, leaving the remaining product, which is commercially known as oleo oil, which, when churned with cream or milk or both and with usually a proportion of creamery butter, the whole being properly salted, gives the new food-product, oleomargarine.

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[1] Science, vol. 7, pp. 471–472.

[2] Senate Mis. Doc. No. 131, Forty-ninth Congress, first session, p. 224.

> In making butterine we use neutral lard, which is made from selected leaf lard in a very similar manner to oleo oil, excepting that no stearine is extracted. This neutral lard is cured in salt brine for forty-eight to seventy hours at an ice-water temperature. It is then taken, and, with the desired proportion of oleo oil and fine butter, is churned with cream and milk, producing an article which, when properly salted and packed, is ready for market.
>
> In both cases coloring matter is used, which is the same as that used by dairymen to color their butter. At certain seasons of the year, viz, in cold weather, a small quantity of salad oil made from cotton seed is used to soften the texture of the product, but this is not generally used by us.

Gustavus F. Swift, of the firm of Swift & Co., of the town of Lake (near Chicago), describes as follows the method in use in the manufacture of artificial butter by his company:[1]

> The fat is taken from the cattle in the process of slaughtering, and after thorough washing is placed in a bath of clean, cold water and surrounded with ice, where it is allowed to remain until all animal heat has been removed. It is then cut into small pieces by machinery and cooked at a temperature of about 150° until the fat in liquid form has separated from the fibrine or tissue; then settled until it is perfectly clear. Then it is drawn into draining vats and allowed to stand a day, when it is ready for the presses. The pressing extracts the stearine, leaving the remaining product, which is commercially known as oleo oil, which, when churned with cream or milk, or both, and with usually a proportion of creamery butter, the whole being properly salted, gives the new food product, oleomargarine.
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> In making butterine we use neutral lard, which is made from selected leaf-lard in a very similar manner to oleo oil, excepting that no stearine is extracted. This neutral lard is cured in salt brine for forty-eight to seventy hours at an ice-water temperature. It is then taken, and with the desired proportion of oleo oil and fine butter, is churned with cream and milk, producing an article which, when properly salted and packed, is ready for market.
>
> In both cases coloring matter is used, which is the same as that used by dairymen to color their butter. At certain seasons of the year, viz, in cold weather, a small quantity of sesame oil, or salad oil made from cotton seed, is used to soften the texture of the product.

WHOLESOMENESS OF ARTIFICIAL BUTTER.

On this subject there is a wide difference of opinion. It is undoubtedly true that a great deal of artificial butter has been thrown upon the market that has been carelessly made, and therefore harmful to the health. On the other hand a butter substitute, made carefully out of the fat of a perfectly healthy bullock or swine, is not prejudicial to health.

Prof. Henry Morton, of the Stevens Institute, Hoboken, N. J., made the following statements before the Senate Committee on Agriculture, pending the consideration of the "Oleomargarine" bill:[2]

> The subject is one which has been of great interest to all scientific men from the time of the original discovery by Mége, which was made, as you are aware, during the siege of Paris. Many persons have been interested in it and have followed it up. I have been frequently called upon to examine processes and superintend operations where modifications in the manufacture have been suggested, and so on, and specimens have been brought to me as a chemist, to examine from time to time microscopic-

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[1] *Op. cit.*, p. 225.

[2] *Op. cit.*, p. 47.

ally and chemically. When the substance was first introduced, the question was raised as to whether it could be distinguished from butter by any test, and I was led in that way to investigate the subject, and to examine as to all the properties which it exhibited, as well as to compare different samples of it, and I have in my experiments in this line examined great numbers of specimens of oleomargarine prepared as butter and of oleomargarine oil for the preparation of butter, from all parts of the country, and also have visited factories very frequently and spent long periods there. I have remained as long as a week in one of these factories continuously—sometimes spending the night as well as the day there, in order to watch the process completely and see the operation from beginning to end, to see what was put in and what was not, and to observe what was done and what was not done.

In the course of these examinations I have reached the conclusion, founded on these observations, that the material is of necessity, a pure one, and cannot possibly be unwholesome, and is, in fact, in that sense a thoroughly desirable and safe article of food. I will express as briefly as I can my reasons for this opinion, and state the facts on which they are founded.

In the first place I have found, as a matter of observation, that fat which is to be used in the manufacture of oleomargarine, if it is in the slightest degree tainted before the manufacture begins, if it is not strictly fresh, if it is not taken almost directly from the slaughtered animal, if it is allowed to stand in a barrel for a few hours in ordinary weather or in cold weather, if put in a barrel with any animal heat in it for a few hours, then an incipient change begins which, in the succeeding process, is exaggerated so that an utterly offensive material is produced, which could not be used for any such purpose.

Prof. C. F. Chandler says:[1]

In all of these reports I have taken the ground that this is a new process for making an old article, and that article is butter. This is a new process for making butter. It is made of materials which are in every respect wholesome and proper articles of food, whether it be made solely from the oleomargarine extracted from beef fat, or whether it has added to it more or less leaf lard properly prepared, or more or less sesame oil or cotton-seed oil, and whether it be or not colored with annatto or the other coloring matters used. I take the ground that there is nothing in any one of these materials in any sense unwholesome, and nothing in any one of them which makes it inferior as an article of food to dairy butter. I regard the discovery of Mége-Mouries, of a process by which beef fat and hog fat can be extracted from adipose tissue and converted into a wholesome article of food free from any disagreeable taste or odor, as one of the most important discoveries made in this century, a discovery by which it is possible to make a perfectly pure and satisfactory, as well as wholesome, article of food at a reasonable price. I have visited various factories where this article is manufactured, from the time the industry began down to date. I am perfectly familiar with the materials employed and the different processes, and know there is nothing whatever used either in material or process which is unwholesome or in any way deleterious to the public health.

Professor Chandler further has reported as follows to the Board of Health of New York City:[2]

NEW YORK, *May* 2, 1881.

To the Board of Health of the Health Department:

Having been directed by this board to investigate the subject of oleomargarine, in response to the resolutions of the Board of Aldermen, I would beg leave to submit the following report:

The resolutions directing the inquiry are as follows:

Whereas there is existing at the present time in the minds of the public great alarm and distrust in relation to the adulteration of food products; and

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[1] *Op. cit.*, p. 67.

[2] *Op. cit.*, p. 70.

Whereas the committee on public health of the assembly of this State has been for some time investigating the adulteration of food products, and especially oleomargarine; and

Whereas this committee have conducted such investigation by calling as witnesses principally dealers in butter and have not examined as witnesses medical or chemical experts to determine the value of oleomargarine as food; therefore

Resolved, That the board of health of this city be, and they are hereby requested and directed to take immediate measures to investigate in the most thorough manner, by medical and chemical aid, the purity, healthfulness, and value of said product as an article of food, and to report to this body the results of their investigation, with such recommendations, if any be necessary, as may relate to the manufacture and distribution of the same as an article of food.

This subject has been before the board on former occasions, and I have little to add to what has been previously stated.

Oleomargarine, invented by the distinguished French chemist, Mége-Mouries, is manufactured in New York City in a few large establishments. The material is fresh beef suet, brought directly from the slaughter-houses. It is thoroughly washed, rendered very carefully, strained to remove a portion of the hard stearine, and then churned with milk to convert it into artificial butter, which contains the same constituents as dairy butter. The process is extremely ingenious and simple and executed by machinery. Nothing objectionable exists in the original material, nor is anything objectionable added during the process, and the operations are conducted with the utmost cleanliness. The product is palatable and wholesome, can be made of uniform quality the year round, is in every respect superior as an article of food to a large proportion of dairy butter sold in this city, and can be manufactured at a much lower price. I regard it as a most valuable article of food and consider it entirely unexceptional in every respect. In this opinion I am supported by the best scientific authorities in the country. The following distinguished chemists, after carefully studying the manufacture, have made the most decided statements in favor of this new article of food:

Prof. George F. Barker, University of Pennsylvania.
Dr. Henry A. Mott, jr., New York.
Prof. G. C. Caldwell, Cornell University, Ithaca, N. Y.
Prof. S. W. Johnson, Yale College, New Haven, Conn.
Prof. C. A. Goessmann, Massachusetts Agricultural College, Amherst, Mass.
Prof. Henry Morton, Stevens Institute, Hoboken, N. J.
Prof. Charles P. Williams, Philadelphia, Pa.
Prof. W. O. Atwater, Wesleyan University, Middletown, Conn.
Prof. J. W. S. Arnold, University of New York.

I would further say that this question is one on which there is no difference of opinion among scientific investigators familiar with the chemistry of dairy products and fats. I have never seen a statement emanating from any person having any standing among scientific men in which a contrary opinion is advanced. There has recently been a very strong confirmation of my opinion published in England. A bill came before the House of Commons in England, directed against this kind of butter from America, and, after considerable discussion, was defeated by a vote of 75 to 59. In the discussion the strongest opponent to legislation against it was Dr. Lyon Playfair, one of the most distinguished chemists and sanitary authorities in England. A pupil of Graham and Leibig, he has filled the chairs of chemistry in the Royal Institution of Manchester and at the University of Edinburgh, was appointed chemist to the Museum of Practical Geology by Sir Robert Peel, represented the universities of Edinburgh and Aberdeen in Parliament, was postmaster-general in the first Gladstone cabinet, has been member of several sanitary commissions, and is now a leading member of Parliament. In his remarks he stated that "bad butter was a fraud upon the poor, and oleomargarine would sooner or later drive it out of the market;" he "thought that good oleomargarine at one shilling a pound was a great deal better and cheaper than bad butter at one shilling four pence a pound," and he said that "as a general rule the former (oleomargarine) did not become so readily rancid as the latter (butter)."

I would further state that, as there is nothing unwholesome in oleomargarine, no legislation in regard to this article is necessary to protect the public health.

C. F. CHANDLER,
President.

Prof. G. F. Barker says:[1]

UNIVERSITY OF PENNSYLVANIA,
Philadelphia, March 22, 1880.

To the United States Dairy Company:

GENTLEMEN: In reply to your inquiry, I would say that I have been acquainted for several years with the discovery of Mége-Mouries for producing butterine from oleomargarine fat. In theory the process should yield a product resembling butter in all essential respects, having identically the same fatty constituents. The butterine prepared under the inventor's patents is, therefore, in my opinion, quite as valuable a nutritive agent as butter itself. In practice the process of manufacture, as I have witnessed it, is conducted with care and great cleanliness. The butterine produced is pure and of excellent quality, is perfectly wholesome, and is desirable as an article of food. I can see no reason why butterine should not be an entirely satisfactory equivalent for ordinary butter, whether considered from the physiological or commercial standpoint.

Prof. G. C. Caldwell, of Cornell University, gives the following testimony:[2]

CHEMICAL LABRATORY, CORNELL UNIVERSITY,
Ithaca, N. Y., March 20, 1880.

I have witnessed, in all its stages, the manufacture of "oleomargarine" and of oleomargarine butter or "butterine."

The process for oleomargarine, when properly conducted, as in the works of the Commercial Manufacturing Company, is cleanly throughout, and includes every reasonable precaution necessary to secure a product entirely free from animal tissue, or any other impurity, and which shall consist of pure fat made up of the fats commonly known as oleine and margarine. It is, when thus prepared, a tasteless and inodorous substance, possessing no qualities whatever that can make it in the least degree unwholesome when used in reasonable quantities as an article of food.

In the manufacture of butterine, since nothing but milk, annotto, and salt, together with, perhaps, a little water from clean ice, are added to this oleomargarine, to be intimately mixed with it by churning and other operations, I have no hesitation in affirming that this also, when properly made according to the Mége patent, and other patents held by the United States Dairy Company, and when used in reasonable quantities, is a perfectly wholesome article of food; and that, while not equal to fine butter in respect to flavor, it nevertheless contains all the essential ingredients of butter, and since it contains a smaller proportion of volatile fats than is found in genuine butter it is, in my opinion, less liable to become rancid.

It cannot enter into competition with fine butter; but in so far as it may serve to drive poor butter out of the market, its manufacture will be a public benefit.

Prof. S. W. Johnson, of Yale College, makes the following statement:[3]

SHEFFIELD SCIENTIFIC SCHOOL OF YALE COLLEGE,
New Haven, Conn., March 20, 1880.

The United States Dairy Company:

GENTLEMEN: I am acquainted with the process discovered by M. Mége for producing the article known in commerce as oleomargarine or butterine.

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[1] *Op. cit.*, p. 73.

[2] *Op. cit.*, p. 73.

[3] *Op. cit.*, p. 74.

I have witnessed the manufacture in all its stages, as carried out on the large scale, and I can assert that when it is conducted according to the specifications of M. Mége it cannot fail to yield a product that is entirely attractive and wholesome as food, and one that is for all ordinary culinary and nutritive purposes the full equivalent of good butter made from cream.

Oleomargarine butter has the closest resemblance to butter made from cream in the external qualities—color, flavor, and texture. It has the same appearance under the microscope, and in chemical composition differs not in the nature, but only in the proportions of its components. It is, therefore, fair to pronounce them essentially identical.

While oleomargarine contains less of those flavoring principles which characterize the choicest butter, it is, perhaps for that very reason, comparatively free from the tendency to change and taint, which speedily renders a large proportion of butter unfit for human food.

I regard the manufacture of oleomargarine or butterine as a legitimate and beneficent industry.

S. W. JOHNSON,
Professor of Theoretical and Agricultural Chemistry,
Director of the Connecticut Agricultural Experiment Station.

Dr. C. A. Goessmann, of Amherst, indorses in general the above statements:[1]

AMHERST, MASS., *March* 20, 1880.

United States Dairy Company, New York:

GENTLEMEN: I have visited on the 17th and 18th of the present month your factory, on West Forty-eighth street, for the purpose of studying your mode of applying Mége's discovery for the manufacture of oleomargarine butter or butterine. A careful examination into the character of the material turned to account, as well as into the details of the entire management of the manufacturing operation, has convinced me that your product is made with care, and furnishes thus a wholesome article of food. Your oleomargarine butter or butterine compares in general appearance and in taste very favorably with the average quality of the better kinds of the dairy butter in our markets. In its composition it resembles that of the ordinary dairy butter; and in its keeping quality, under corresponding circumstances, I believe it will surpass the former, for it contains a smaller percentage of those constituents (glycerides of volatile acids) which, in the main, cause the well-known rancid taste and odor of a stored butter.

I am, very respectfully, yours,

C. A. GOESSMANN, PH. D.,
Professor of Chemistry.

To these I may add the names of Prof. Charles P. Williams, of the State University of Missouri, Dr. Henry Mott, jr., Prof. W. O. Atwater, and Prof. J. W. S. Arnold.[2]

Armsby[3] says in respect of the healthfulness of oleomargarine:

Very exaggerated and absurd statements have been made regarding the unhealthfulness of butterine and oleomargarine. The charges have in general been that the fat used is practically uncooked, and that raw animal fat is unwholesome; that filthy fat and fat from diseased animals are used, and that the product contains, or is liable to contain, the germs of disease; and that in cleansing these diseased and filthy fats dangerous chemicals are used, which are not subsequently completely removed.

That the fats used are of themselves unwholesome there is no proof whatever. They contain nothing that butter-fat does not also contain, and differ from it only by the absence of about 6 per cent. of the glyceride of certain soluble fatty acids, viz, caprinic,

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[1] *Op. cit.*, p. 74.

[2] *Op. cit.*, pp. 73, 74, 75.

[3] Science, vol. 7, No. 172.

caprylic, capronic, and butyric acids. The only experiments upon the digestibility of imitation butter are two, by A. A. Mayer, upon oleomargarine. These showed a difference of only about 2 per cent. in favor of butter. That the higher flavor of butter acting upon the nervous system would give it a greater nutritive value than the flavorless "neutral" or "oleo" may be conceded; but that an article which even experts fail to distinguish from genuine butter is at any serious disadvantage in this respect may well be doubted.

The manufacturers claim that imitation butter can only be made from the best quality of fat from freshly-killed animals, and I know of no evidence which disproves their assertions. The sensational article recently published in a prominent agricultural paper in the Northwest, accompanied by cuts of the numerous organisms found in butterine, is of no significance in this connection, both because the species described are all harmless, and because no comparative examinations of genuine butter were made. It is highly probable that many samples of the latter would show as miscellaneous an assortment of formidable looking, harmless organisms as did the butterine.

On the other hand, however, there is at present no guaranty, except the statement of the manufacturers, that diseased fat is not or cannot be used, the manufacture being conducted entirely without any official inspection, and visitors being in most (not all) cases excluded. I believe that the chances of disease being conveyed in this way are small, but they are not yet proved to be non-existent.

As regards filthy processes of manufacture, it may safely be asserted that butterine could not successfully imitate butter were it not as clean as most things are which pass for clean in this dirty world.

The charge that dangerous chemicals are used in the manufacture may be disposed of in a few words. If a dangerous amount of any chemical which is claimed to be used were left in the finished product the latter would be inedible. Should trace of these chemicals be found their significance would not lie in themselves, but in the indication they would furnish that the original fats were impure and required chemical treatment.

Sell[1] has made an examination of the evidence for and against the unwholesomeness of artificial butter and has reached the following conclusions:

The artificial butter prepared from the fat of healthy animals, apart from possibly a somewhat less digestibility, in comparison with milk-butter furnishes in general no reason for the supposition that it can affect injuriously human health.

There is ground for the suspicion that a part of the artificial butter occurring in commerce is manufactured out of such material or by such processes as do not with certainty exclude the danger of conveying to man disease whether produced by vegetable spores or animal parasites.

There is ground for suspicion that a part of the artificial butter is made from nauseating substances.

The possibility of injury to health from a carelessly-prepared artificial butter must not be neglected.

Dr. Thomas Taylor presented this aspect of the case to the Senate Committee.[2]

It has already been mentioned that in the earlier processes employed in the manufacture of artificial butter the stomachs of sheep and pigs were digested with the fats employed.

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[1] Arbeiten a. d. Kaiserlichen Gesundheitsamte, pp. 494, 500.

[2] *Op. cit.* pp. 42–46 and 273–4.

Tidy and Wigner[1] have investigated the action of mammary tissue on fats used as butter substitutes.

By digesting a pure animal fat with the chopped-up tissues of the udders of cows the authors found a marked chemical change produced. Oleomargarine or tallow when treated in this way give rise to both soluble and volatile fatty acids. Since both milk and butter contain a certain amount of mammary tissue, in the form of casts from the mammary glands, it is believed that they also would exert an influence on animal fats. Butter appears to act more vigorously than milk in this way, probably because it contains a larger percentage of mammary tissue.

NUTRITIVE VALUES OF BUTTER AND OLEOMARGARINES.

On this subject Atwater[2] has collected valuable information, he says:

> The value of butter, as of any other food material for nourishment, depends upon the amounts of its nutritive ingredients, their digestibility, and their uses in the nutrition of the body.
>
> ### CHEMICAL COMPOSITION.
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> The food values of real and imitation butter, as compared with each other and with other food materials, can be best shown by first comparing their composition.
>
> It appears that the nutrients of the leaner kinds of meat and fish consist mostly of protein, that the fatter meats and fish contain considerable fat with the protein, that the vegetable foods have for the most part very little fat, and abound especially in carbohydrates, while the nutriments of butter and oleomargarine consist almost exclusively of fats. Indeed, the protein and carbohydrates in both must be regarded as impurities. The quantities of fat are shown by analysis to be very nearly the same in both.
>
> ### DIGESTIBILITY.
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> Regarding the relative digestibility of butter and oleomargarine the experimental facts at hand are meager. They imply, as would be expected from the composition, that there is very little difference between the two. The study of the question is rendered difficult by the fact that what is ordinarily called the digestibility of a food includes several different things, the ease with which it is digested, the time required for digesting it, and the proportions of its several constituents that are digested.
>
> As to the comparative ease and time of digestion of butter and oleomargarine nothing is definitely known, though there is little ground for assuming that, in the alimentary canal of a healthy person, at any rate, one would be digested and taken into the circulation much more readily than the other. The actual amounts digested are capable of more nearly accurate experimental estimate. During the past few years very many experiments have been made, in Germany especially, to test the quantities of the more important constituents of different foods digested by domestic animals, and a considerable number have been carried out with men and children.
>
> The only comparative experiments on the digestibility of butter and oleomargarine that have been reported are two series conducted by Professor Mayer, a German chemist. One series was with a full-grown man and the other with a boy of nine years of age, both strong, healthy persons. The outcome was that both the man and the boy digested from 97.7 to 98.4 per cent. of the fat of the butter, and from 96.1 to 96.3 per cent. of the fat of the oleomargarine. The average difference was about 1.6 per cent. in favor of the butter. There are, however, certain unavoidable sources of

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[1] Analyst, 1883, pp. 113 *et seq.*

[2] Bradstreet's, Saturday, June 19, 1886.

error in such experiments, and it is very probable that the proportions actually digested were somewhat larger than these figures imply. Very likely each of the two persons may have digested practically all of the fat of the butter, and all but 1 or 2 per cent. or even less of that of the oleomargarine. In these experiments the butter and oleomargarine were eaten with bread, cheese, white of eggs, potatoes, peas, and sugar.

The digestibility of butter has been tested in two or three other series of experiments. Thus Dr. Rubner, in Munich, found that a healthy man, on a diet of butter, bread, and meat, digested 97.3 per cent. of the total fat of the food, of which the bulk came from the butter. In some experiments by myself, in which a man received a diet of fish (haddock) and butter, 91 per cent. of the total fat, nearly all of which came from the butter, was found to be digested.

The experiments of Rubner and myself were conducted in the same manner as those of Mayer, and exposed to the same slight sources of error. The results of all of them are just what would naturally be expected, namely, that very nearly all of the fat of butter and of oleomargarine is digested in a healthy organism.

It might seem that the relative digestibility of the two materials could be tested by experiments in artificial digestion; that is to say, by treating both substances with digestive fluids, or with materials similar to them, and observing the results. Such experiments are not accurate tests of the actual digestibility of the substances in the body, since the conditions which obtain in the alimentary canal cannot be exactly imitated by any artificial means which physiological chemistry has yet suggested. Professor Mayer, taking into account that the fats are more or less split up in the process of natural digestion, has made some experiments to test the comparative readiness with which butter and oleomargarine are split up, and finds a very slight difference in favor of butter. As the result of all his experiments he concludes that, while the butter appears to be a little more digestible than oleomargarine, the difference is too small to be of practical consequence for healthy persons. At the same time there may be cases, especially those of invalids and children just past the nursing period, when butter would be preferable; but, considering simply the nutritive values for ordinary use, Professor Mayer considers the choice between the two to be essentially one of comparative cost, an opinion from which there is, so far as I am aware, scarcely any dissent among those who have devoted the most study to this class of subjects.

It is a common and perhaps correct theory, though it lacks experimental confirmation, that the flavor of the fats peculiar to butter may in some way increase its value for nutriment. But, granting this to be true, it would be hardly reasonable to assume that a difference in flavor which even experts may fail to detect could make any considerable difference in the nutritive effect of two substances otherwise so similar as real and imitation butter.

To recapitulate briefly, butter and oleomargarine have very nearly the same chemical composition; in digestibility there may be a slight balance in favor of butter, though for the nourishment of healthy persons this difference can hardly be of any considerable consequence; for supplying the body with heat and muscular energy, which is their chief use in nutrition, they are of practically equal value, excelling in this respect all other common food materials. Such, at any rate, is the practically unanimous testimony of the latest and best experimental research.

While it is true that chemical analysis and certain digestive experiments have not hitherto shown that pure butter possesses any marked superiority over butter surrogates as a food, yet it must not be forgotten that butter has a much more complex composition than lard or tallow or cotton-seed oil; that it is a natural food, and doubtless possesses many digestive advantages which science has not yet been able to demonstrate.

THE MANUFACTURE OF ARTIFICIAL BUTTER IN THE UNITED STATES.

The following information has been kindly furnished by the Hon. Joseph S. Miller, Commissioner of Internal Revenue:

TREASURY DEPARTMENT, OFFICE OF INTERNAL REVENUE,
Washington, March 4, 1887.

SIR: In reply to your letter of 1st instant, I have the honor to state that there are thirty-seven factories engaged in the manufacture of artificial butter now in operation in the United States, located as follows:

Location.	No. of factories.	Location.	No. of factories.
Denver, Col	2	Buffalo, N. Y	1
Chicago, Ill	11	Columbus, Ohio	1
Kokomo, Ind	1	Cleveland, Ohio	3
Kansas City, Kans	1	Philadelphia, Pa	3
Armourdale, Kans	1	Pittsburgh, Pa	1
Cambridge, Mass	1	Allegheny, Pa	1
Brooklyn, N. Y	2	Providence, R. I	3
New York, N. Y	3	Pawtucket, R. I	2

There are two hundred and fifty-nine wholesale dealers in the United States, located as follows:

Location.	No. of wholesale dealers.	Location.	No. of wholesale dealers.
Birmingham, Ala	2	Salt Lake, Utah	1
Fort Smith, Ark	1	Hoboken, N. J	1
Hot Springs, Ark	1	Jersey City, N. J	1
Little Rock, Ark	2	Deming, N. Mex	1
Pine Bluff, Ark	1	Fairbanks, Ariz	1
Denver, Col	4	Saratoga Springs, N. Y	1
Jacksonville, Fla	1	Rochester, N. Y	1
Atlanta, Ga	1	Youngstown, Ohio	2
Englewood, Ill	1	Philadelphia, Pa	12
Springfield, Ill	1	Allegheny City, Pa	1
Cairo, Ill	2	Woonsocket, R. I	1
Council Bluffs, Iowa	1	Memphis, Tenn	8
Elwood, Kans	1	El Paso, Tex	2
Louisville, Ky	3	Dallas, Tex	1
New Orleans, La	5	Dennison, Tex	1
Boston, Mass	30	Milwaukee, Wis	3
Fall River, Mass	1	Ashland, Wis	1
Lowell, Mass	3	Eau Claire, Wis	1
Aspen, Col	1	Worcester, Mass	3
Pueblo, Col	1	Houghton, Mich	1
Durango, Col	1	Detroit, Mich	6
Buena Vista, Col	1	Grayling, Mich	1
Hartford, Conn	2	Saginaw, Mich	1
New Haven, Conn	4	Luddington, Mich	1
Leadville, Col	4	Saint Paul, Minn	2
Chicago, Ill	21	Helena, Mont	2
Peoria, Ill	1	Jefferson City, Mont	1
Danville, Ill	1	South Butte, Mont	1
Indianapolis, Ind	1	Ogden, Utah	1
Kansas City, Kans	2	Dover, N. H	1
Topeka, Kans	1	Newark, N. J	1
Covington, Ky	1	Sante Fé, N. Mex	1
Baltimore, Md	2	Albuquerque, N. Mex	1
Salem, Mass	1	New York, N. Y	15
New Bedford, Mass	2	Buffalo, N. Y	1
Gloucester, Mass	2	Cincinnati, Ohio	6
Lawrence, Mass	2	Cleveland, Ohio	3
Springfield, Mass	1	Pittsburgh, Pa	14
Ironwood Mich	1	Providence, R. I	16
Bay City, Mich	3	Pawtucket, R. I	1
East Saginaw, Mich	1	Nashville, Tenn	1
Muskegon, Mich	1	San Antonio, Tex	1
Grand Rapids, Mich	4	Fort Worth, Tex	2
East Saint Louis, Ill	1	Richmond, Va	1
Butte, Mont	2	Oshkosh, Wis	1
Missoula, Mont	1	Hurley, Wis	2
Omaha, Nebr	3	Chippewa Falls, Wis	1

The quantity manufactured and removed for consumption or sale at 2 cents per pound during the months of November and December, 1886, and January, 1887, is as follows:

	Pounds.
November	4,742,569
December	2,786,278
January	2,501,114
Total	10,029,961

The quantity exported from the United States during the period above, all exportations being from the port of New York, is as follows:

	Pounds.
November	3,247
December	58,689
January	52,761
Total	114,697

Respectfully,

JOS. S. MILLER,
Commissioner.

Hon. N. J. COLMAN,
Commissioner of Agriculture, Washington, D. C.

COLORING MATTERS IN BUTTER.

The pure animal fats, prepared in the manner described, are almost colorless. The tint of genuine butter is imparted to these bodies by various coloring matters. The principal artificial colors which have been employed are:

Annotto (*Bixa orellana*).
Turmeric (*Curcuma longa* and *viridiflora*).
Saffron (dried stigmas *Crocus sativus*).
Marigold leaves (*Calendula officinalis*).
Yellow wood (*Morus tinctoria*).
Carrot juice (*Daucus carota*).
Chrome yellow ($PbCrO_4$)
Dinitrocressol—kalium.

ANNOTTO.

This substance is used more than any other in imparting to artificial butter a yellow tint. Indeed it is used to color genuine butter, which often in winter is almost white in its natural state.

The coloring substance called *annotto, arnatta,* or *roucou* is the reddish pulp surrounding the seeds in the fruit of *Bixa orellana,* a middling-sized tree growing in Guiana and other parts of South America. The pulp is separated by bruising the fruit, mixing it with water, then straining through a sieve, and allowing the liquid to stand till the undissolved portion subsides. The water is then poured off and the mass which remains, having been sufficiently dried, is formed into flat cakes or cylindrical rolls and sent into the market. Another mode is to bruise the seeds, mix them with water, and allow the mixture to ferment. The coloring matter is deposited during the fermentation, after which it is removed and dried. In commerce there are two kinds of annotto the Spanish or Brazilian and French, the former coming in baskets from Brazil, the latter in casks from French Guiana. The French, which is also called *flag annotto*, has a disagreeable smell, probably from having been prepared by the fermenting process, but is superior as a dye-stuff to the Spanish, which is without any disagreeable odor. Annotto is of a brownish red color, usually rather soft but hard and brittle when dry, of a dull fracture, of a sweetish peculiar odor, and a rough, saline, bitterish taste. It is inflammable, but does not melt with heat. It softens in water, to which it imparts a yellow color, but does not dissolve. Alcohol, ether, the oils, and alkaline solutions dissolve the greater part of it. It contains a peculiar crystallizable coloring principle, to which M. Preisser, its discoverer, gave the name of *bixin*. It is frequently adulterated with red ocher, powdered bricks, colcothar, farinaceous substances, chalk, sulphate of calcium, turmeric, &c. The mineral substances, if present, will be left behind when the annotto is burned.[1]

SAFFRON.

Saffron has a peculiar, sweetish, aromatic odor, a warm, pungent, bitter taste, and a rich deep orange color, which it imparts to the saliva when chewed. The stigmas of which it consists are an inch or more in length, expanded and notched at the upper extremity, and narrowing towards the lower, where they terminate in a slender, capillary, yellowish portion, forming a part of the style. When chewed it tinges the saliva deep orange-yellow. Saffron should not be mixed with the yellow styles. When pressed between filtering paper it should not leave an oily stain. When soaked in water it colors the liquid orange-yellow, and should not deposit any pulverulent mineral matter nor show the presence of organic substances differing in shape from that described.[2]

Adulteration of saffron.—Saffron is often adulterated with cheaper yellow vegetable coloring matter, turmeric, annotto, the flowers of the marigold (*Calendula officinalis*), Carthamus flowers, the flowers of *Arnica montana, Scolymus hispanicus, Puticaria dysenterica, Punica granatum, Pæonia, Crocus vernus*, &c.[3]

Of these the marigold flowers are perhaps the most commonly used. They have a natural yellow color, and when they are saturated with carmine or aniline red, and dried, they possess a striking similarity to the genuine saffron. If they are put for a few minutes in water, however, they assume their original form, and are then easily distinguished from the stigmas of the saffron flower.

If a mixture of saffron stigmas and the substitutes just mentioned be put into a vessel of water where the individual pieces are widely separated, the saffron stigmas soon become surrounded with a yellow extract, while the others suffer no change or impart only a weak carmine tint to the water.

The use of mineral coloring matters like the chromate of lead is highly reprehensible from a sanitary point of view.

Annotto and saffron in butter may be detected by the following method, proposed by Cornwall:[4]

About 5 grams of the warm filtered fat are dissolved in about 50cc. of ordinary ether, in a wide tube, and the solution is vigorously shaken for ten to fifteen seconds with 12 to 15cc. of a very dilute solution of caustic potash or soda in water, only alkaline enough to give a distinct reaction with turmeric paper, and to remain alkaline after separating from the ethereal fat solution. The corked tube is set aside and in a few

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[1] U. S. Dispensatory, p. 1572.

[2] U. S. Dispensatory, p. 501.

[3] Schimpfer, Anleit. z. Mikroskopischen Untersuchung d. Nahrungs- und Genussmittel, p. 101.

[4] Chem. News, vol. 55, p. 49.

hours, at most, the greater part of the aqueous solution, now colored more or less yellow by the annotto, can be drawn from beneath the other with a pipette or by a stopcock below, in a sufficiently clear state to be evaporated to dryness and tested in the usual way with a drop of concentrated sulphuric acid.

Sometimes it is well to further purify the aqueous solution by shaking it with some fresh ether before evaporating it, and any fat globules that may float on its surface during evaporation should be removed by touching them with a slip of filter-paper; but the solution should not be filtered, because the filter-paper may retain much of the coloring matter.

The dry-yellow or slightly orange residue turns blue or violet blue with sulphuric acid, then quickly green, and finally brownish or somewhat violet (this final change being variable, according to the purity of the extract).

Saffron can be extracted in the same way; it differs from annotto very decidedly, the most important difference being in the absence of the green coloration.

Genuine butter, free from foreign coloring matter, imparts at most a very pale yellow color to the alkaline solution; but it is important to note that a mere green coloration of the dry residue on addition of sulphuric acid is not a certain indication of annotto (as some books state) because the writer has thus obtained from genuine butter, free from foreign coloring matter, a dirty green coloration, but not preceded by any blue or violet blue tint.

Blank tests should be made with the ether; it is easy to obtain ether that leaves nothing to be desired as to purity.

Turmeric is easily identified by the brownish to reddish stratum that forms between the ethereal fat solution and the alkaline solution before they are intimately mixed. It may be even better recognized by carefully bringing a feebly alkaline solution of ammonia in alcohol beneath the ethereal fat solution with a pipette, and gently agitating the two, so as to mix them partially.

Martin[1] gives a method of separating and determining artificial coloring matters in butter. To 5 grams of fat, dry, are added 25cc. CS_2 and the mixture well shaken with water made slightly alkaline with NaOH or KOH and the mixture gently shaken. The alkaline water will dissolve all the coloring matter. This is now determined qualitatively by the spectroscope or quantitatively by making up a comparative mixture with the coloring matter found. Butters act better when treated as above than oleomargarine.

The relative amount of color in butters is thus estimated by Babcock:[2]

The relative amount of color in butters may be determined with accuracy as follows: One gram of the fresh butter is digested with 15cc. of refined kerosene till the fats are completely dissolved and the solutions filtered. The filtrate will be colored in proportion to the coloring matter of the butter, and may be compared to that from another butter or preferably to a standard solution by means of a Dubosque colorimeter. A standard color for comparison may be prepared by adding a small quantity of any of the commercial butter colors to kerosene oil. This standard will keep for a long time without changing, if kept from the light.

The scale of the colorimeter on the side which the butter solution occupies is always set at the same degree, while the scale for the other standard is made variable. The reading of this side will, therefore, vary with the amount of color in the sample.

If some of the kerosene oil in which the butters are dissolved be substituted for the solution of butter, a small reading will be obtained which should be deducted

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[1] Analyst, 1885, p. 163.

[2] Fifth An. Rep't B'd Control, N. Y. Exp. Sta., p. 335–336.

from that for each of the butters. The numbers remaining are directly proportional to the colors of the butters. In the butters thus far examined a fair colored Jersey butter was taken for a standard and called 100. The others were calculated to this standard from the scale reading.

The use of a small amount of vegetable coloring matters mentioned above does not seem to be prejudicial to health.

EXAMINATION OF BUTTERS.

The examination of butters to detect adulterations may be divided into two parts: (1) Determination of physical properties; (2) determination of chemical properties.

Physical properties.—The physical properties of fats which are useful in butter analysis are their crystalline state, specific gravity, and melting point.

Pure fresh butter prepared in the ordinary manner is not crystalline. The microscope shows the absence of all forms of crystalline structure, and thin films of the butter fat have no influence whatever on polarized light.

On the contrary, old butters, or butters which have been melted and allowed to crystallize, and oils and fats which have been once in a fluid state, show, as solids, quite a distinct crystalline structure readily revealed by the microscope and affecting, in a marked manner, the polarized ray.

Recently much attention has been excited by a discussion of the application of polarized light to the qualitative examination of suspected butters, and since many analysts have not the time to fully investigate this matter I have thought it useful to enter upon the discussion of it in considerable detail.

Polarization is a term applied to a phenomenon of light, in which the vibrations of the ether are supposed to be restricted to a particular form of an ellipse whose axes remain fixed in direction. If the ellipse becomes a straight line it is called "plane polarization." This well-known phenomenon is most easily produced by a Nicol prism, consisting of a crystal of carbonate of calcium (Iceland spar). This rhombohedral crystal, the natural ends of which form angles of 71° and 109°, respectively, with the opposite edges of its principal section, is prepared as follows:

The ends of the crystal are ground until the angles just mentioned become 68° and 112°. The crystal is then divided diagonally at right angles with the planes of the ends and with the principal section, and after the new surfaces are polished they are joined again by Canada balsam. The principal section of this prism passes through the shorter diagonal of the two rhombic ends. If now a ray of light fall on one of the ends of this prism, parallel with the edge of its longer side, it suffers double refraction, and each ray is plane polarized, the one at right angles with the other. That part of the entering ray of light which is most refracted is called the ordinary and the other the extraordinary ray. The refractive index of the film of balsam being intermediate between those of the rays, permits the total reflection of the ordinary ray, which, passing to the blackened sides of the prism, is absorbed. The extraordinary ray passes the film of balsam without deviation and emerges from the prism in a direction parallel with the incident ray, having, however, only half of its luminous intensity.

Two such prisms, properly mounted, furnish the essential parts of a polarizing apparatus. They are called the "polarizer" and the "analyzer," respectively.

If now the plane of vibration in each prism be regarded as coincident with its principal section, the following phenomena are observed: If the prisms are so placed that the principal sections lie in the prolongation of the same plane, then the extraordinary polarized ray from the polarizer passes into the analyzer, which practically may be regarded in this position as a continuation of the same prism. It happens, therefore, that the extraordinary polarized ray passes through the analyzer exactly as it did through the polarizer, and is not reflected by the film of balsam, but emerges from the analyzer in seemingly the same condition as from the polarizer. If now the analyzer be rotated 180°, bringing the principal section again in the same plane, the same phenomenon is observed. But if the rotation be in either direction only 90°, then the polarized ray from the first prism, incident on the second, deports itself exactly as the ordinary ray, and on meeting the film of balsam is totally reflected. The field of vision, therefore, is perfectly dark.

In all other inclinations of the planes of the principal sections of the two prisms the ray incident in the analyzer is separated into two, an ordinary and extraordinary, varying in luminous intensity in proportion to the square of the cosine of the angle of the two planes.

Thus by gradually turning the analyzer, the field of vision passes slowly from maximum luminosity to complete obscurity.

The expression "crossed Nicols" refers to the latter condition of the field of vision.

Selenite plate.—In the practical application of polarized light to the examination of facts, an important use is made of a selenite plate (crystallized sulphate of calcium). A disk of selenite, interposed between the polarizer and analyzer imparts a coloration to the field of vision which varies with the relative position of the principal sections of the two prisms.

This phenomenon depends on the fact that a plane polarized ray of light can be decomposed, in passing a section of a bi-refracting crystal like selenite or mica, into two rays, polarized at right angles and differing in phase.

This fact is illustrated by passing a polarized ray (from a Nicol prism) through a very thin crystallized plate of mica or gypsum (selenite) obtained by cleavage. By the double refraction of the thin plate the polarized ray is separated into two, ordinary and extraordinary. The extraordinary, having to pass over a greater distance, joints the ordinary ray, after emergence, with a phase slightly different, the degree of difference depending on the nature of the lamina, the inclination of the incident ray, &c., but in every case this difference of phase can be easily calculated, and the resultant beam of light is said to be elliptically polarized. Each of the components of this ray enters the analyzer and is again resolved. One of its elements is suppressed in the Nicol and the other, consisting of vibrations in the principal plane, passes through. The result is two sets of vibrations in the same plane slightly different in phase, which are, therefore, in a condition to interfere and produce color. If the source of light be monochromatic, when the analyzer is rotated, only certain variations in luminous intensity will be observed; but if, on the other hand, white light be employed these variations in phase will give rise to a display of colors. In order that the field of vision be of a uniform tint it is necessary that the lamina of crystal be of uniform thickness. For ordinary use the selenite plate is ground to a thickness which will give green and red tints.

For crossed Nicols the colors of the selenite plate appear brightest when it is so placed that the plane of vibration in the crystal forms an angle of 45° with the plane of vibrations of the polarized incident ray. If the selenite plate is rotated in its own plane, the color appears in the four quadrants at its maximum and disappears at intervals of 90°.

If the planes of the two Nicols are parallel, the same order of phenomena appear as before, except that the positions of maximum and minimum are reversed.

If the analyzer be rotated and the selenite plate and polarizer remain stationary there is no effect produced, when the principal section of the selenite is parallel or perpendicular to the polarizing plane of the under Nicol. But if this plane is inclined less than 45° to that of the polarizer, then the selenite plate in a complete revolution of the analyzer will appear four times brightly colored and four times colorless. In adjoining quadrants the colors will be complementary. When the Nicols are so placed as to produce the maximum intensity of color, if small bi-refracting crystals be introduced at random into the field of vision, they will, in general, have the same effect on the plane polarized ray as the selenite plate. Since the axes of these crystals may have any accidental position with reference to the planes of the Nicols, it follows that the field of vision, which before appeared of a uniform tint, will now become variegated, the color disappearing in some cases and becoming more intense in others.

When a bi-refracting crystal is cut into laminæ normal to its axis, of appropriate thickness, it gives some peculiar phenomena when examined with polarized light. When the analyzer is perpendicular to the polarizer, there is seen in the ordinary image a black cross, the existence of which can be explained by the mathematical theory of polarization. The arms of this cross are parallel and perpendicular to the primitive plane of polarization. Between the arms are generally to be found rings which present the successive tints of the fringes of interference. In the extraordinary image the order of the phenomenon is entirely reversed.

Having now briefly described the more important optical phenomenon which forms the basis of the examination of butters with polarized light, I will next say something of the nature of the substances to be examined.

The expressions "fats" and "oils" designate those natural products of animals and vegetables known as glycerides. Chemically considered they are the normal propenyl ethers of the fatty acids, or, in other words, compounds of the triad alcohol, glycerine, with the fatty acids. The term "fat" is applied to such bodies when they are solid at ordinary temperatures, and "oil" when they are semi-solid or liquid. Those which are most important are:

Tri-stearin, $C_3H_5(C_{18}H_{35}O_2)_3$, occurs in natural fats. It may be obtained in a considerable degree of purity by repeated crystallizations from ether. It crystallizes in plates of a pearly luster. Its melting point is 55° C.

Tri-palmitin, $C_3H_5(C_{16}H_{31}O_2)_3$, is found in animal fats and palm oil. It crystallizes with a pearly luster from ether. The crystals have a melting point of from 50° to 66° C.

Tri-butyrin, $C_3H_5(C_4H_7O_2)_3$, occurs chiefly in butter. At ordinary temperature it is liquid, and has a distinct and peculiar odor and taste.

Tri-olein, $C_3H_5(C_{18}H_{33}O_2)_3$, occurs in animal fats and in almond and olive oil. At ordinary temperatures it is liquid, is neutral to test papers, and has neither taste nor smell.

Minute quantities of tri-myristin, caprin, caprylin, and caproin are also found in butter.

Pure butter fat is supposed to contain—

	Per cent.
Tri-olein, about	42.5
Tri-stearin, about	51.0
Tri-butyrin, about	6.3
Other glycerides, about	.2
	100.00

Olive oil is composed chiefly of tri-palmitin and olein.

Tri-stearin is the chief constituent of mutton fat, it having only small quantities of olein and palmitin.

Beef fat has somewhat more palmitin and stearin than mutton tallow. Lard has more olein.

It is thus seen that in dealing with butter fats and their substitutes we have to consider chiefly tri-olein and stearin, and, in smaller quantities, tri-palmitin, butyrin, &c. It follows, therefore, that the chief differences in the several substances will be due to the different proportions in which these glycerides are mixed and to such other physical differences as the various sources of the substances under examination would produce. These differences, however, prove greater when subjected to physical and chemical analysis than the foregoing résumé of their chemical constitution would indicate. Advantage has been taken of these differences of physical structure to discriminate between fats and oils of different origins. The specific gravity and the melting point furnish two valuable points of discrimination, but both of these are perhaps inferior in value to the evidence afforded by the crystalline structure of the fats. The observation with the microscope of the crystals obtained in various ways furnishes valuable data for discrimination, and if the light employed be plane polarized or elliptically polarized by a selenite plate, these data become still more valuable.

The first account of the use of the selenite plate in such examinations was given by Dr. J. Campbell Brown in the Chemical News, vol. 28, pages 1, *et seq.* He gives the following directions for the polaro-microscopic work:

> Examine several portions of the original sample by means of a good microscope, using a one-fourth or one-fifth inch object-glass. In butter made from milk or cream nothing is seen except the characteristic globules, and the granular masses of curd and the cubical crystals of salt. The hard fats of butter are present in the globules in a state of solution, and are not recognizable in a separate form.
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> If stearic acid, stearin, or palmitin be present in separate form, they will be recognizable by simple fusiform crystals, or starlike aggregations of acicular crystals. They indicate the presence of melted fats.
>
> Other substances, such as starch flour, palm oil, corpuscles, Irish moss, coloring matter, &c., may also be distinguished by the microscope as distinct from butter or fats.
>
> Examine the same portions with the same object-glass, together with a polariscope, consisting of two Nicol's prisms and a selenite plate. The crystals referred to polarize light, and when viewed by the polariscope are distinctly defined. Particles of suet and other fats which have not been melted may also be distinguished by their action on polarized light, by their amorphous form, and by their membranes.

The value of this deportment of fresh-butter fat with elliptically polarized light did not meet with the appreciation its merits deserved until attention was again called to it by Prof. Thomas Taylor, of the Department of Agriculture.

Any fat or oil which is homogeneous and non crystalline will present the same phenomena when viewed with polarized light and selenite plate; in other words will have no effect on the appearance of the field of vision. It is only, therefore, fats which are in a crystalline or semi-crystalline state that can thus be distinguished from fresh, amorphous butter. Naturally it follows that a butter which has been melted and cooled, or butter which has stood a long time, would impart a mottled appearance to the field of vision. For a simple preliminary test, however, the procedure is worthy of more attention than its discoverer, Dr. J. Campbell Brown, accorded to it.

FORMS OF FAT CRYSTALS.

The forms of fat crystals differ greatly with the kinds of fat and the proportions in which they are mixed. It would be idle to attempt a description of all these modifications.

Husson[1] has published an illustrated description of some of the more important fat crystals. Suet crystals, according to Husson, are very characteristic of stearin. They are small, rounded, or elliptical masses formed by stiff, needle-like crystals, and resemble a sea urchin or hedge hog.

In lard are seen polyhedral cells arising from the compression of the fatty globules. In impure lard are also seen the remains of cells and adipose tissue. Fresh butter shows some long and delicate needles of margarine (?) united in bundles and grouped in various ways. When the butter is melted these needles diminish in length and become grouped round a central point. I have mentioned these descriptions especially for the purpose of calling attention to the fact, that, in the illustrations of the microscopic appearance of butter and other fats, emphasis is often given to one particular phenomenon, and the real appearance as seen in the microscope is not reproduced.

The only reliable representation is found in the actnal photo-micrograph or its exact graphic reproduction.

When the crystals of certain fats are prepared in a special way they show, with polarized light, a distinct cross, the existence of which is explained by the laws of elliptical polarization already mentioned.

This cross was first described by Messrs. Hehner and Angell in 1874 in the following words:

> If some of a fat containing crystals be placed on a slide and a drop of castor or olive oil be applied and pressed out with a thin glass cover, the depolarization of light is much enhanced; a revolving black cross, not unlike that on some starch grains, is seen in great perfection. These crosses are most clearly defined in the crystals obtained from butter, and these thus mounted form a brilliant polariscopic object.

They add further:

> Thus far and no farther, as it seems to us, can the microscope assist us in this matter; but even such indications are valuable, especially when subsequent analysis proves the sample to be an adulterated article. The microscopic evidence in such case frequently serves to clinch together the whole superstructure, and thus certainty is made doubly sure.

Dr. Thomas Taylor has further called attention to this phenomenon in a paper read before the American Society of Microscopists at its Cleveland meeting, August, 1885. On page 3 of the reprint of this paper he says:

> Since the publication of that paper I have experimented largely with butter, and have made the discovery that when it is boiled and cooled slowly for a period of from twelve to twenty-four hours at a temperature of from 50° to 70° Fahr. it not only becomes crystallized, but, with proper mounting and the use of polarized light, it ex-

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[1] Ann. d. Chem. et d. Pharm., ~~vol. 5, pp. 12~~, 469.

> hibits on each crystal a well-defined figure resembling what is known as the cross of Saint Andrew. In course of time, the period ranging from a few days to a few weeks according to the quality of the butter used and the temperature to which it is exposed, the crystals, which at first are globular, degenerate, giving way to numerous rosette-like forms peculiar to butter.

On page 5 he says:

> About ten years ago, while making some experiments with boiled butter, I first observed it exhibited small crystals somewhat stellar in form, but gave no further attention to the fact until May last. For the purpose of determining the real form of the crystal of boiled butter I procured a sample of pure dairy butter from Ohio. I boiled it, and when cold examined it under a power of 75 diameters. To my surprise I found globular bodies. When I subjected them to polarized light a cross consisting of arms of equal length was observed on each crystal. On rotating the polarizer the cross of each crystal rotated. On rotating the glass on which the specimen of butter was mounted the crosses remained stationary, thus showing that the appearance of the cross depends, probably, on the fact that the crystals are (1) globular, (2) polarizing bodies, (3) translucent, and (4) comparatively smooth. Were they opaque or non-polarized or did they consist of long spines, causing great divergence of the rays of light, no image of the cross would be visible; showing that the appearance of the cross under polarized light and the conditions stated is not due to any physical structure of the fatty crystals themselves. But from whatever cause the appearance of the cross on the butter crystals arises, its constant appearance on new butter under the conditions above described is a fact beyond any question; and, as far as my experience goes, the better the quality of the butter the more clearly defined is the cross; it is black, large, and well defined. When these crystals are under polarized light and a selenite plate combined they exhibit the prismatic colors, but the cross proper is not visible in this case, although the crystals are still divided into four equal parts and are exceedingly interesting objects.

Dr. Taylor having thus directed the attention of scientists to these important phenomena, it has not taken long to show that there is little reason for the rather mean opinion of European chemists of the value of the microscope in detecting adulterations of butter. In several cases of prosecution before the District authorities the offenders have been convicted solely on the microscopical evidence and have admitted the justice of the sentence. If only fresh butters were exposed for sale, and all adulterants were certainly once melted and slowly cooled, but little more than this qualitative examination would be necessary.

Prof. H. A. Weber, of Columbus, Ohio, has made some interesting experiments with the microscope on fats, which in the main bear out the conclusions of Messrs. Brown, Hehner and Angell, and Taylor. As was to be expected, however, he has shown that the appearance of the cross on a crystal of natural fat does not show that it is derived from pure butter. He says, in Bulletin No. 13 of the Ohio Experimental Station, Experiments 7, 8, 9, and 10:

> *Experiment* 7.- The difference between the behavior of the tallow fats in Experiment 3 and the ast three experiments could only be ascribed to a difference of conditions. It is well known that table butter normally contains 4 to 6 per cent. of salt and 5 to 20 per cent. of water. These ingredients constitute the most marked difference between butter and the rendered animal fats as tallow and lard. In order to test the effect of this mixture upon the tallow fats, about half an ounce of the oleo oil used in Experiment 3 was mixed in a porcelain mortar with a small quantity of

salt and eight or ten drops of water. After the water was thoroughly incorporated, the mass was transferred to a test tube and boiled for 1 minute as in the case of butter. It was then poured into a wooden pill-box and allowed to cool as before. The cooled mass presented quite a marked difference in appearance from that obtained from the same substance in Experiment 3. It retained to a great extent the yellow color of the oleo oil, was of a more granular nature, and in fact resembled boiled butter in every respect. When a small particle was stirred up with olive oil on a glass slide it separated readily. When covered and viewed with a pocket lens it revealed a mass of globules resembling insect eggs. Under the microscope these globules exhibited essentially the same characteristics as those obtained from butter in Experiment 1. The crystalline mass of the oleo globule seemed somewhat coarser, and to this condition was ascribed the fact that the cross, as well as the colors produced by the selenite plate, were less sharply defined than in the globules obtained from butter. The slides prepared from this material were remarkably free from the small detached crystals of fat observed in Experiment 3.

Experiment 8.—Having thus discovered that these globular masses may be obtained from pure tallow fat by simply observing the conditions which obtain in butter making, the following test was made: Nine grams of oleo oil and 1 gram of lard were placed in a small beaker glass and eight or ten drops of a saturated solution of salt in water added. The mixture was then gently heated to melt the fats. After shaking violently for a few moments to mix the salt solution with the fats, the mixture was boiled gently for 1 minute and then allowed to cool as before in a wooden pill-box. The microscopic examination of this preparation revealed globular masses which could in no wise be distinguished from those obtained from pure butter. The crystalline texture was dense, the cross of St. Andrew's plainly marked, and the colors produced by the selenite sharply defined.

Experiment 9.—A mixture of one part of lard to five parts of oleo oil was treated as in the last experiment with like results.

Experiment 10.—In this test a mixture consisting of 20 per cent. of lard and 80 per cent. of oleo oil was employed. Whether the consistency of this mixture was peculiarly adapted to the formation of the globules, or whether possible variations of conditions in manipulation were more favorable, the writer is unable to judge from a single experiment, but the fact is that in this case the individual "butter crystals" were exceedingly large and characteristic.

The use of polarized light in photo-micrography is also valuable in enabling the photographer to print the light-colored crystals on a dark background. To illustrate some of the forms of crystals of butter and its substitutes as they appear under polarized light a large number of microscopic samples were prepared and photographed by Messrs. Richards and Richardson. Results of some of the more interesting of these photographs are herewith transmitted. In all cases the figures are magnified 40 diameters, unless otherwise stated.

DESCRIPTION OF PLATES.

PLATE I.

FIG. 1.—Fresh butter boiled.

FIG. 2.—Fresh butter made in the laboratory without the use of salt, melted, filtered, and boiled. A small sample of the butter was taken and boiled for one minute in a test tube over the naked flame, then set aside and allowed to cool slowly for twenty-four hours. A suitable quantity was then taken, sufficient to make a slide for the microscope, thinned with olive oil, and pressed out on the cover.

PLATE II.

FIG. 3.—Fresh Virginia butter boiled and let stand for seven days. Specimen prepared by Dr. T. Taylor, March 11, 1886.

FIG. 4.—Fresh Virginia butter boiled. Specimen prepared by Dr. T. Taylor March 11, 1886.

PLATE III.

FIG. 5.—Fresh Kentucky butter boiled. Specimen prepared by Dr. T. Taylor March 11, 1886.

FIG. 6.—Fresh butter boiled. Specimen prepared by Dr. T. Taylor March 15, 1886.

PLATE IV.

FIG. 7.—Filtered butter fat dissolved in boiling alcohol and allowed to cool slowly.

FIG. 8.—Filtered butter fat dissolved in boiling ether and allowed to cool slowly. The fresh butter was melted and filtered through a jacketed filter, thus getting rid of the water, curd, and salt; allowed to cool and prepared as above.

PLATE V.

FIG. 9.—Beef suet fat. Not boiled. The suet fat was cut up into fine pieces and melted in the water bath at a low heat and filtered; allowed to cool slowly. Specimen was taken several days after the sample was prepared.

FIG. 10.—Beef suet fat boiled with the addition of salt and cooled slowly.

PLATE VI.

FIG. 11.—Beef suet fat, "oleo oil," dissolved in boiling ether and allowed to cool slowly.

FIG. 12.—Beef suet fat, "oleo oil," dissolved in boiling alcohol and allowed to cool slowly.

PLATE VII.

FIG. 13.—Leaf lard. Not boiled. Specimen taken direct from can as purchased in the open market. Magnified 160 diameters.

FIG. 14.—Lard dissolved in boiling ether and allowed to cool slowly.

PLATE VIII.

FIG. 15.—Beef fat, "oleo oil," and lard, "neutral," boiled with salt and water and allowed to cool slowly.

FIG. 16.—Butterine, from Armour & Co., Chicago. Boiled and allowed to cool slowly.

PLATE IX.

FIG. 17.—Butterine, from Armour & Co., Chicago. Not boiled. Specimen taken direct from tub as received July, 1886. Magnified 160 diameters.

FIG. 18.—Butterine, from Armour & Co., Chicago. Melted, filtered, and boiled.

PLATE X.

FIG. 19.—Butterine, from Armour & Co., Chicago. Dissolved in boiling ether and allowed to cool slowly.

FIG. 20.—Oleo oil, from Armour & Co., Chicago. Melted, filtered, and boiled. Allowed to stand four days under cover glass.

PLATE XI.

FIG. 21.—Oleomargarine, from Armour & Co., Chicago. Not boiled. Specimen taken direct from tub as received July, 1886. Magnified 160 diameters.

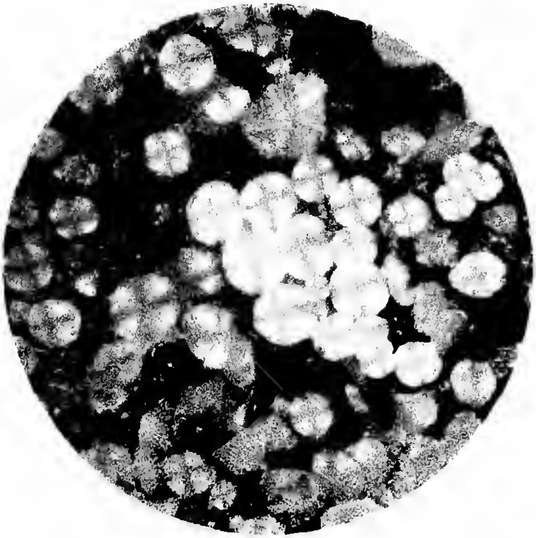
FIG. 22.—Oleomargarine, from Armour & Co., Chicago. Boiled with salt and water and allowed to cool slowly.

PLATE XII.

FIG. 23.—Same as Fig. 22.

FIG. 24.—Same as Fig. 22.

Fig 1



BUTTER x40

Fig 2

BUTTER x40

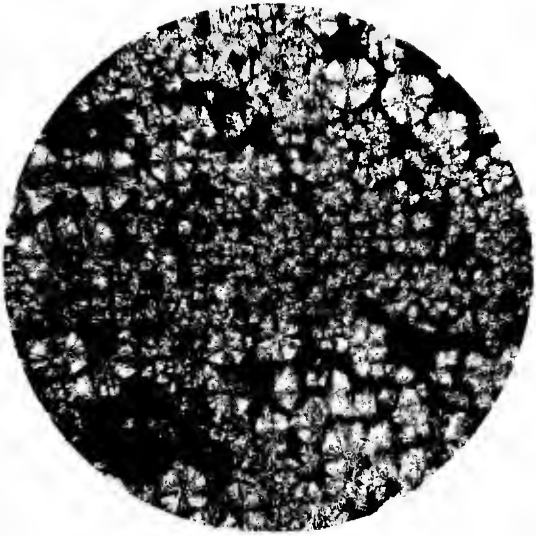
Photo. by Clifford Richardson.

A. Hoen & Co. Heliocaustic. Baltimore.

Fig 3

BUTTER x40

Fig 4



BUTTER x40

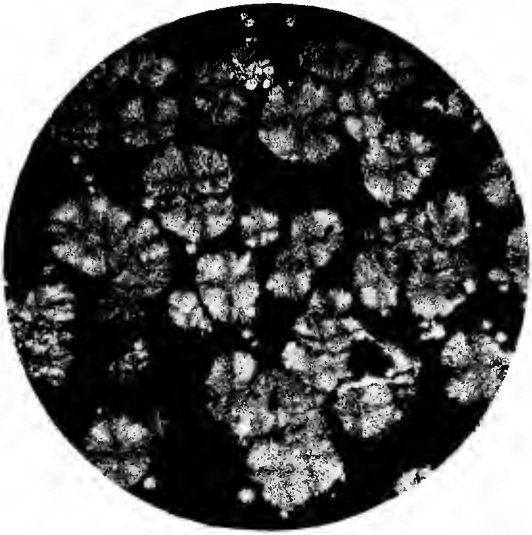
Photo. by Clifford Richardson.

A. Hoen & Co. Heliocaustic Baltimore.

Fig 5

BUTTER x40

Fig 6

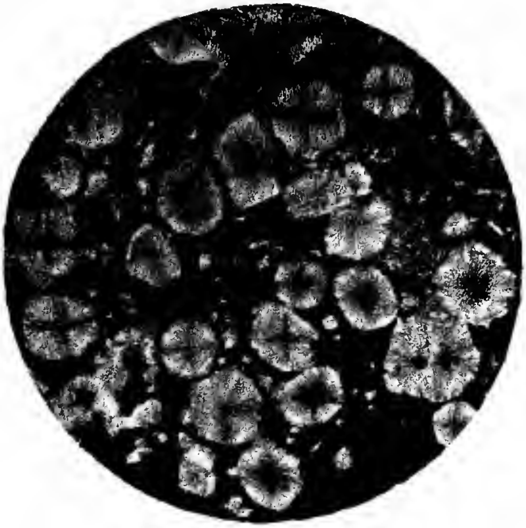


BUTTER x40

Photo. by Clifford Richardson.

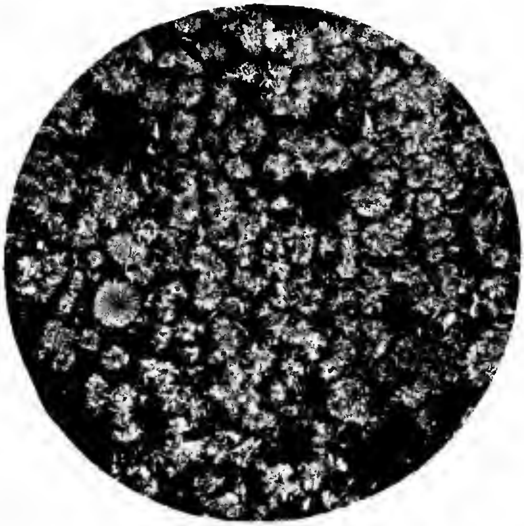
A Hoen & Co. Heliocaustic Baltimore

Fig 7



BUTTER x40

Fig 8



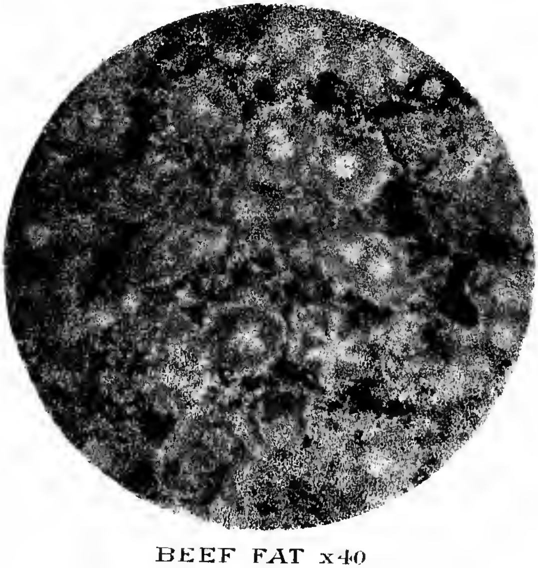
BUTTER x40

Photo. by Clifford Richardson. A. Hoen & Co. Helioaustic Baltimore

Fig 9

BEEF FAT x40

Fig 10

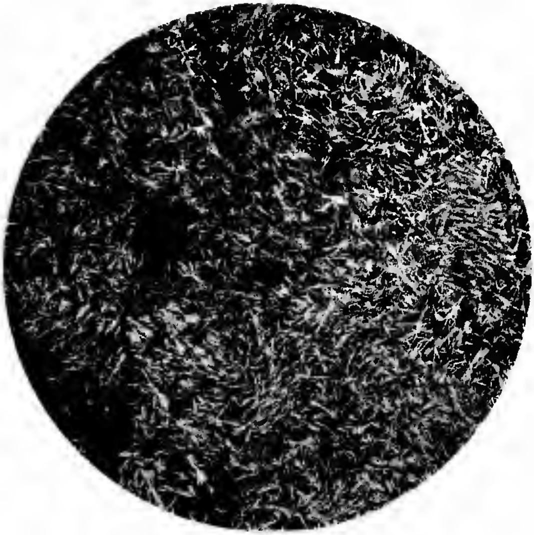


BEEF FAT x40

Photo. by Clifford Richardson.

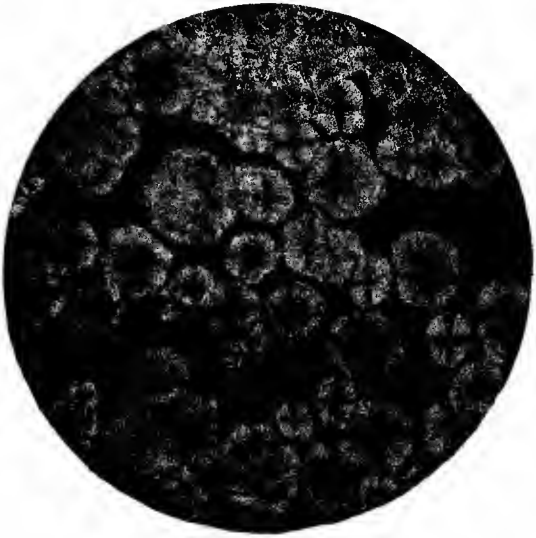
A. Hoen & Co. Heliocaustic, Baltimore

Fig 11



BEEF FAT x40

Fig 12

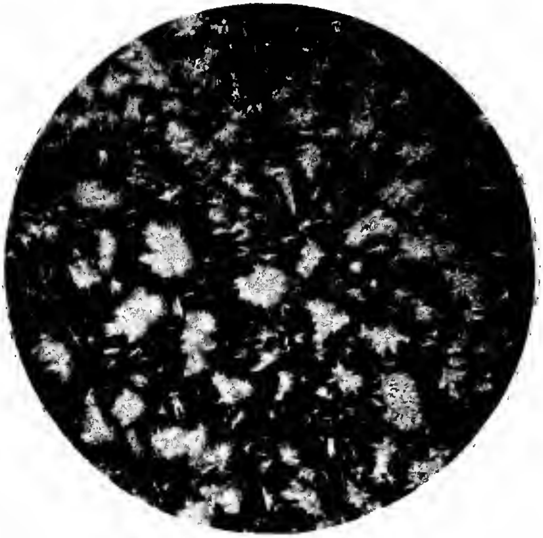


BEEF FAT x40

Photo. by Clifford Richardson.

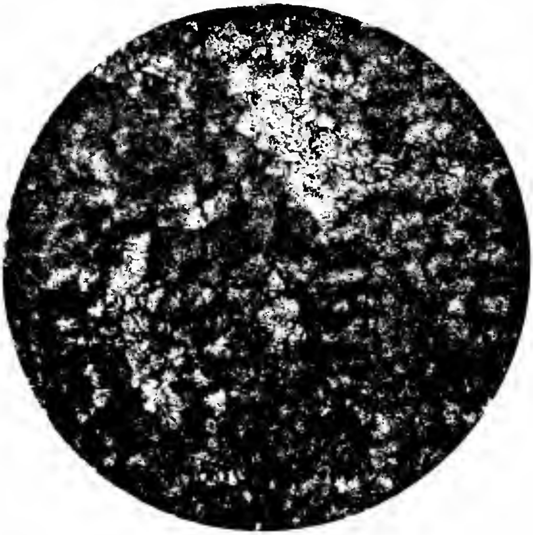
A. Hoen & Co. Heliocaustic, Baltimore.

Fig 13



LARD x160

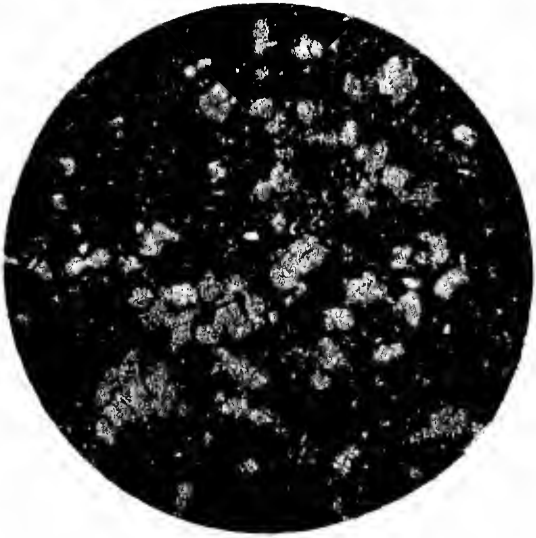
Fig 14



LARD x40

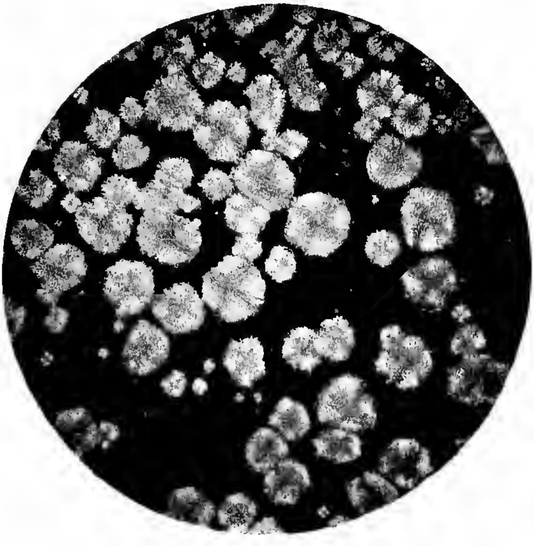
Photo. by Clifford Richardson. A. Hoen & Co. Heliocaustic, Baltimore.

Fig 15



BEEF FAT & LARD x 40

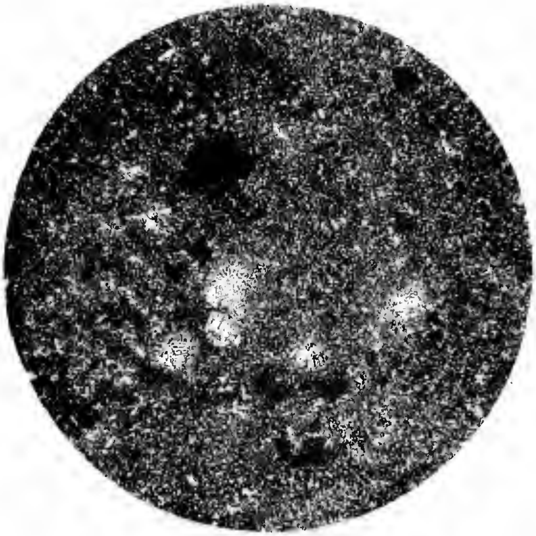
Fig 16



BUTTERINE x 40

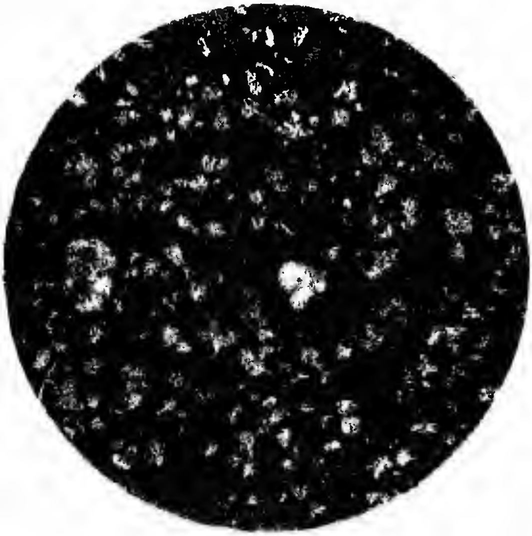
Photo. by Clifford Richardson. A. Hoen & Co. Heliocaustic, Baltimore

Fig 17



BUTTERINE x160

Fig 18

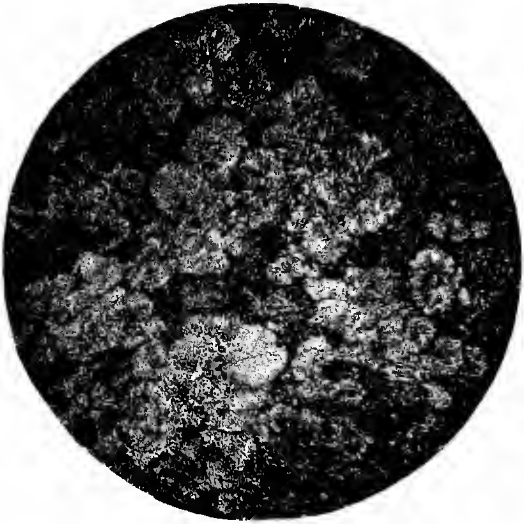


BUTTERINE x40

Photo. by Clifford Richardson.

A. Hoen & Co. Heliocaustic Baltimore.

Fig 19



BUTTERINE x 40

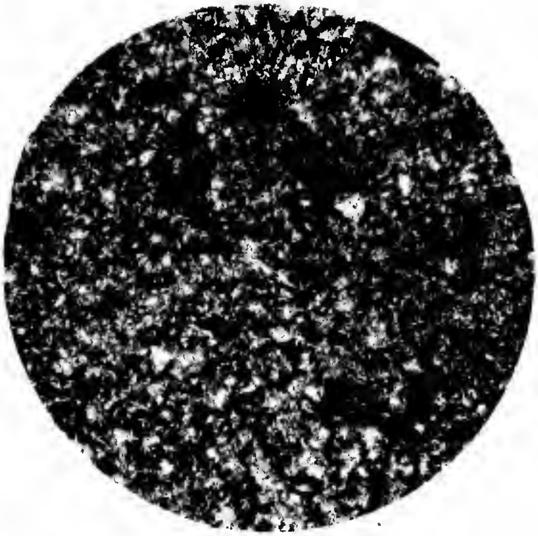
Fig 20

BEEF FAT x 40

Photo. by Clifford Richardson.

A. Hoen & Co Heliocaustic, Baltimore.

Fig 21



OLEOMARGARINE x160

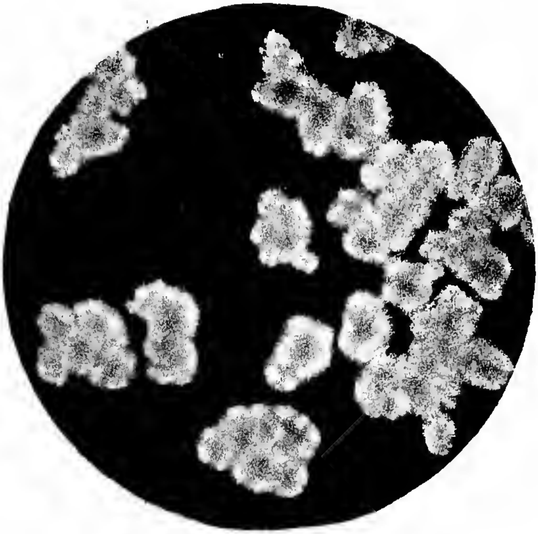
Fig 22

OLEOMARGARINE x40

Photo. by Clifford Richardson.

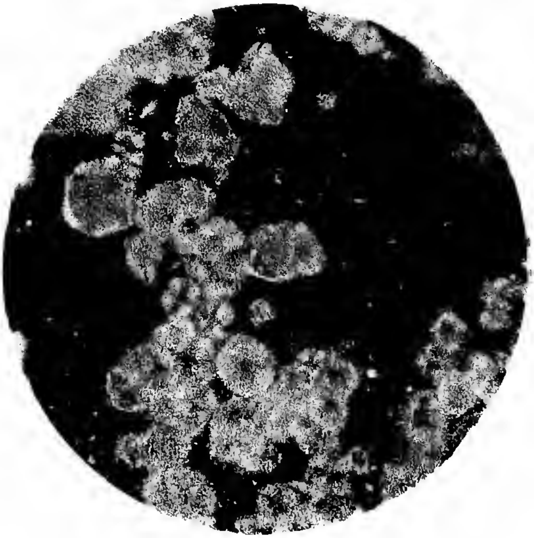
A Hoen & Co Heliocaustic, Baltimore.

Fig 23



OLEOMARGARINE x 40

Fig 24



OLEOMARGARINE x 40

Photo. by Clifford Richardson. A. Hoen & Co Heliocaustic, Baltimore.

A careful study of these illustrations will show that the microscope and polarized light are most valuable and reasonably certain means whereby a qualitative examination of butters can be made. The approximate amount of added fat can only be determined by a chemical analysis of the suspected sample, taken in connection with some of its further physical characteristics.

I have given above all the really valuable points heretofore established in respect of the use of polarized light in butter and fat analyses.[1]

The use of the microscope in butter examinations has not commanded as much attention among analysts as its merits deserve. Sell says:[2]

> Though investigations have shown that the differences in structure under the microscope are not in all cases sufficiently characteristic to determine a sharp distinction between two different fats, yet it must be admitted that the microscopic examination is able to prove the presence of foreign fats at the moment it succeeds in establishing the presence of molecular tissues in animal or vegetable parasites.

Leudtner and Hilger say:[3]

> The use of the microscope in the examination of fats requires a still further development before it can become generally applicable.

Having written to the editor of the Analyst for some information on the subject of the use of the microscopic methods in England, he replied: "The whole subject has been studied over and there is nothing in it."

Dr. S. M. Babcock, chemist of the New York Experimental Station, says:[4]

> At the time these butters were received there was considerable controversy regarding the efficiency of Dr. Taylor's method for the detection of adulterations in butter by means of the microscope. An excellent opportunity was offered in these samples for testing this method in an impartial manner, and a microscopical examination of them was made before the nature of the butters was revealed by other tests. The butters were examined directly with polarized light and a selenite plate, and afterwards the crystals from the melted butters were examined in the same way for the "Saint Andrew's cross."
>
> The direct examination with polarized light and a selenite plate showed prismatic colors in all of the adulterated butters, and a uniform tint in all of the genuine butters, except No. 2, which appeared very much like the adulterated samples. The crystals from all of the butters, adulterated as well as genuine, gave a well-defined Saint Andrew's cross with polarized light. This was also the case with neutral lard (No. 14), in which the cross was sharply defined, though quite small. No. 15 consisted of stearine from the oleo-oil factories, and showed no cross when examined by itself, but when combined with a small quantity of butter fat the crystals formed had the same appearance as those from pure butter.
>
> The method has also been quite unsatisfactory in trials made at the station with butters whose character was known. Whether these results were due to a lack of skill or to imperfect knowledge in the details of the work I do not know. The

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[1] Notices of minor importance on the same subject can be found in Chem. News, vol. 4, pp. 230, 283, 309, 322; Zeit. Anal. Chem., 1872, p. 334; Journ. Royal Microscopical Society, 1878, p. 378; American Quarterly Microscopical Journal, 1878, p. 294; American Journal of Microscopy, October, 1878; Bied. Centralblatt, 1879, pp. 861–865; 1882, p. 345; 1882, p. 49; Amer. Chemist, vol. 2, p. 428.

[2] *Op. cit.*, p. 503.

[3] Ver. Bay. Vertreter d. Angewand. Chem., p. 222.

[4] Fifth Ann. Rept. Bd. Control N. Y. Exp. Sta., pp. 330, 331.

uncertain results of some skilled microscopists, however, would indicate that the difficulty is inherent in the method. It certainly is not simple, and is not calculated to supersede the chemical methods now in use.

Caldwell,[1] after references to the notices of the use of the microscope in the examination of butter published up to that time (1882), says, p. 519:

> It is plain, therefore, that little dependence can be placed on any microscopic test of the genuineness of butter, at least so far as the observation of the crystalline forms of foreign fats is concerned, for neither does the absence of such forms prove that the butter does not contain oleomargarine, nor does their presence prove the adulteration.

On the other hand, Mylius[2] has shown that the polarization microscope may be used for the detection of minute quantities of foreign fats in butter. Pure butter gives with crossed Nicols a dark field, whereas crystals of foreign fat will appear bright. Skalweit[3] recommends this method highly, and affirms that even the kind of foreign fat present may be determined.

In spite of the generally unfavorable opinions I feel sure that the chemist who neglects to make a simple microscopic examination of a suspected butter with polarized light and a selenite plate loses a valuable qualitative indication of the character of the samples with which he has to work. The melting of the sample of butter and its slow cooling to secure good bi-refracting crystals I consider a much less valuable indication than the simple observations above described.

SPECIFIC GRAVITY.

The determination of the specific gravity of a butter fat gives a most valuable indication of its purity. The density of pure butter glycerides is distinctly greater than that of the common adulterants, with the exception of cotton-seed oil. While this difference is not great, it is nevertheless large enough to be easily detected by careful manipulation.

Manipulation.—The relative weight of the filtered and dried fat is to be determined in a picnometer. This flask should be carefully calibrated by weighing the pure distilled water it will contain at the temperature at which the subsequent determinations are to be made. The flask should be provided with a delicate thermometer, but this is not essential, since the temperature can be determined by an external thermometer.

The temperature at which the determinations should be made is evidently that at which all the common butter adulterants will be in a perfectly fluid state. Generally the temperature of 100° F. has been employed. Since, however, "neutral lard" may have a melting point as high as 40° C. or even a little above that I have uniformly taken the specific gravity at that degree. In case the fat should have a melting point a little above this the temperature can be raised until the fat is

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[1] Second Ann. Rept. N. Y. S. Bd. of Health.

[2] Correspondensblatt des Vereins Anal. Chem., 1878, No. 3.

[3] *Ibid.*, 1879, Nos. 5 and 13.

fluid and can then be reduced to 40° C., without danger of solidification. The difference between the specific gravities expressed at 37° C. and 40° C. is not of very great magnitude.

Blyth[1] recommends the use of a picnometer of 50 to 100 grams capacity, with a thermometer stopper. This is filled with water at 35° C. and placed in a beaker of water at 43° C. When the water has reached a temperature of 37°.7 C. the flask is removed and weighed.

The fat whose density is to be determined is treated in the same manner and weighed at the same temperature.

Wigner[2] places the butter-fat in a wide tube where a bubble of the specific gravity of .896 is kept below the surface by the bulb of the thermometer. At a certain temperature the bubbles will slowly sink to the bottom. In butters of .911 density, above which a sample may be passed as pure, these beads will sink as follows:

Specific gravity of beads	.889	.8896
Temperature	62°.7 C.	55°.5 C.

If the beads sink at any temperature lower than these the butter will need further examination.

Estcourt[3] describes a method of taking specific gravity of fats as follows:

The bulb of a Westphal balance is suspended in a test-tube containing the fat, the test-tube being immersed in paraffin, in a water bath. The adjustment of the weights takes place at a temperature of 92°.2 C. This process has been modified by König.[4]

In König's process there are several water baths which are closed with the exception of a tube for carrying off the steam. In the cover of each bath are four openings for the reception of four test-tubes 1¼ inches in diameter and 8 to 9 inches long. These are fastened air-tight into the openings mentioned. Each tube stands one-half inch above the cover of the water bath. Each piece of apparatus when in use contains in one of the tubes a sample of pure butter and in the others the samples under examination.

The specific gravity is determined by small areometers 6 inches long and with a scale marked from .845 to .870. The numbers obtained at 100° C. were as follows: Pure butter, .867; artificial butter, .859; beef fat, .860; mutton fat, .860; lard, .861; horse fat, .861. Mixture of pure butter with other fats gave numbers between .859 and .865.

The process of König has been tested by the Board of Health at Berlin and found relatively useful.[5] The method has also been approved by Elsner;[6] by Ambuhl and Dietzsch;[7] and Meyer.[8]

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[1] Foods, p. 295.

[2] Blyth, *op. cit.*, p. 295.

[3] Chem. News, vol. 34, p. 254.

[4] Industrieblätter, 1879, p. 455.

[5] Sell, *op. cit.*, p. 505.

[6] Die Praxis des Nahrungsmittelchemikers, 2d ed., p. 50.

[7] Rep. d. Ver. Anal. Chem., 1884, p. 359.

[8] Zeit. Anal. Chem., 1881, p. 376.

Jones[1] calls attention to the fact that the specific gravity of butter increases with age.

The specific gravities of several samples are compared in the following table, the numbers in second column being obtained after eighteen months:

Specific gravity at 37.7 *C.*

Sample.	1.	2.
1	.9123	.9083
2	.9105	.9114
3	.9119	.9185
4	.9112	.9165
5	.9125	.9155
6	.9133	.9132

In other samples there was a decrease in specific gravity. In five samples out of nine there was an increase and the percentage of soluble acids had also increased.

Since butters in general are obtained for analysis without having been long kept the observation of Jones does not have much practical importance.

Sendtner and Hilger[2] find that a filtered pure butter fat does not show a specific gravity less than .866 at 100° C. In the Erlangen University numerous experiments with twenty different samples of butter showed variations from .866 to .8685.

Allen[3] recommends Sprengel's tube for the determination of specific gravity of oils at the temperature of boiling water.

The weight of the Sprengel tube and that of water contained in it at 15°.5 C. being known, the tube should be completely filled with the oil by immersing one of the orifices in the liquid and gently sucking the air from the other orifice of the tube. The tube is then placed in the mouth of a conical flask containing water kept in a rapid ebullition, and the cover of a porcelain crucible placed over it. As the oil gets hot it expands and is expelled in drops from the horizontal capillary orifice of the tubes. When the expansion ceases any oil adhering to the orifice is removed by cautious application of filter paper, the tube removed from the bath, wiped dry, allowed to cool, and weighed. The weight of the contents divided by the weight of water at 15°.5 C. previously known to be contained by the tube will give the density of the oil at the temperature of the boiling water; water at 15°.5 C. being taken as unity.

Bendikt[4] prefers the Sprengel tube to all the other methods of estimating the specific gravity of oils. He also recommends the Westphal balance as used by Bell and Walkenhaar.

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[1] Analyst, 1879, p. 39.

[2] Vereinbarungen betreffs d. Untersuch. u. Beurteilung v. Nahrungs-Genussmitteln, pp. 221–2.

[3] Com. Organic Analysis, Vol. 2, 2d ed., p. 15.

[4] Analyse der Fette, &c., p. 53.

Dr. Muter[1] gives the following table of the specific gravity of various oils at 37°.7 C.

Kind of oil.	Specific gravity.	Kind of oil.	Specific gravity.
Olive oil	907.0	Linseed oil (boiled)	938.0
Almond oil	905.6	Castor oil	955.8
Arachis oil	908.5	Sperm oil	872.4
Rape oil	906.7	Whale oil	906.0
Nut oil	908.4	Seal oil	915.0
Cotton-seed oil (brown)	917.6	Codliver oil	917.9
Cotton-seed oil (refined)	913.6	Lard oil	907.8
Poppy-seed oil	915.4	Neat's-foot oil	907.0
Hempseed oil	919.3	Butterine	903 to 906.0
Linseed oil (raw)	925.2	Butter-fat	012 to 914.0

METHOD EMPLOYED IN CHEMICAL DIVISION.

When convenient the determination of specific gravity is not made until a number of samples is on hand. Each determination is made in duplicate. The picnometers, holding about 25 grams, are filled with the filtered fat, at as low a temperature as possible, and placed in a flat dish filled with water as nearly to the tops of the flasks as possible. The picnometers used should all be of the same height. The stopper has a capillary perforation for the escape of the oil as the temperature rises. If the picnometers are not furnished with thermometers of their own, a delicate thermometer is suspended in the water surrounding them. The water-bath is slowly warmed and gently but constantly stirred until the temperature reaches 40° C. It is kept at this temperature for fifteen or twenty minutes, until the fat has taken on the same temperature as the water. The picnometers are then carefully cleaned and dried, and, after cooling to the temperature of the balance-room, are weighed. This method is somewhat tedious when only one determination is to be made, but where many samples are to be examined it is sufficiently speedy. In respect of accuracy it leaves nothing to be desired.

TEMPERATURE AT WHICH SPECIFIC GRAVITY IS STATED.

Different analysts select different temperatures for determining specific gravity. It would be well to have some agreement on this point to avoid confusion.

Since the specific gravity determined at any temperature can be easily calculated for any other given temperature, I suggest that it might be well to express all specific gravities in terms of water at 4° C.

THE MELTING POINT OF FATS.

The fats pass rather slowly from the semi-solid state, which is their natural condition at ordinary temperatures, to complete fluidity. It is, therefore, difficult to determine accurately the exact temperature at which they melt.

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[1] Allen, *op. cit.*, p. 15, foot-note.

The value of the melting point in the examination of fats is at once apparent, provided it is possible to be assured that it represents a definite temperature which can be easily and accurately determined.

At a temperature of 40° C. pure butter fat has a specific gravity of .912, while the substitutes therefor, viz, lard, tallow, oleo-oil, neutral lard, &c., have specific gravities varying from .900 to .905. Yet even these small differences are extremely valuable in distinguishing the fats from each other.

The differences in melting points, when they can be accurately determined, will also prove helpful to the analyst. The usual methods employed to determine melting points have been based on the assumption that a fat becomes transparent at the moment it assumes the liquid state. Usually the fat is melted and placed in glass capillary tubes, and, after cooling, put into water near the bulb of a thermometer. The water is slowly warmed, and the moment the fat in the tube becomes transparent the reading of the thermometer is taken. A careful observer is able in this way to make multiple determinations which agree well together, but the readings of different persons are apt to vary greatly. Moreover, it is not the *melting* but the *transparent* point that is determined.

In 1883, at the Minneapolis meeting of the Association for the Advancement of Science, I described a method of determining the flowing point of a fat. The melted fat having been put into a small bent metallic tube, was, after cooling, placed in a bath of mercury. One arm of this U-tube was slightly longer than the other. The bent tube was immersed in the mercury until the longer arm was just below the surface. The fat in the tube was, therefore, subjected to a certain definite pressure from the mercury, due to the difference in length of the two arms. When the melted fat first appeared on the surface of the mercury, the thermometric reading was made. It is scarcely necessary to add that the bulb of the thermometer was wholly immersed in the mercury. Fairly good results were obtained by this method.

Another method, which gave rather good results, I tried at the same time. A thin film of fat was spread over the surface of the mercury and the temperature noted at which a platinum wire drawn through it left no trace. The solidifying point was determined in the same operation by observing where the wire left a mark. Various methods for determining the melting point of fats are given by Reichert.[1] The method preferred by him is a modification of Guichard's process,[2] in which the fat is forced out of the tube by a water pressure of a constant magnitude.

Dr. H. Krüss[3] describes an apparatus for estimating the melting point by the completion of an electric circuit dependent on the melting of the fat used as an insulating material. A platinum wire, bent into the

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[1] Zeit. Anal. Chem., 1885, pp. 11 *et seq.*

[2] *Ibid.*, 1883, p. 70.

[3] Zeit. f. Instrumentenkunde, vol. 4, pp. 32, 33.

form of a small hook, is dipped into the melted fat, a portion of which adheres to it. This process is repeated until a sufficient insulation is produced. The fat-covered end of the wire is then dipped into a mercury cup, which contains also the bulb of the thermometer. The cup is placed in the electric circuit and the moment of contact is determined by the ringing of an electric bell. Thorough trial of this method convinced me that it was less accurate than any of those which have already been mentioned.

Realizing the importance of determining some definite point at which fats would assume a constant condition under the influence of temperature, I was led to select another physical aspect of fats, easily and certainly visible, which could be regarded as the melting point. This condition may be defined as the point at which the molecular attraction of the fat becomes greater than the molecular cohesion.

If a thin film of any fat be suspended in a liquid of equal specific gravity with it and this liquid be slowly warmed, a point will be reached at which the film will roll up and finally assume the form of a sphere. By imparting to the globule a gentle motion of rotation the observer is easily able to distinguish the moment when it becomes sensibly symmetrical. I use the following method and apparatus for applying this principle to the determination of the melting points of fats.[1] The apparatus consists of (1) an accurate thermometer for reading easily tenths of a degree; (2) a less accurate thermometer for measuring the temperature of water in the large beaker glass; (3) a tall beaker glass, 35cm. high and 10cm. in diameter; (4) a test tube 30cm. high and 3.5cm. in diameter; (5) a stand for supporting the apparatus; (6) some method of stirring the water in the beaker. I use a blowing bulb of rubber and a bent glass tube extending to near the bottom of the beaker; (7) a mixture of alcohol and water of the same specific gravity as the fat to be examined.

Manipulation.—The disks of the fat are prepared as follows: The melted and filtered fat is allowed to fall from a dropping tube from a height of 15 to 20cm. onto a smooth piece of ice floating in water. The disks thus formed are from 1 to 1½cm. in diameter and weigh about 200 milligrams. By pressing the ice under the water the disks are made to float on the surface, whence they are easily removed with a steel spatula.

The mixture of alcohol and water is prepared by boiling distilled water and 95 per cent. alcohol for ten minutes to remove the gases which they may hold in solution. While still hot the water is poured into the test-tube already described until it is nearly half full. The test tube is then filled with the hot alcohol. It should be poured in gently down the side of the inclined tube to avoid too much mixing. If the tube is not filled until the water has cooled the mixture will contain so many air bubbles as to be unfit for use. These bubbles will gather on

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[1] Journal Anal. Chemistry, vol. 1, No. 1, pp. 39 *et seq.*

the disk of fat as the temperature rises and finally force it to the top of the mixture.

The test tube containing the alcohol and water is placed in a vessel containing cold water, and the whole cooled to below 10° C. The disk of fat is dropped into the tube from the spatula, and at once sinks until it reaches a part of the tube where the density of the alcohol-water is exactly equivalent to its own. Here it remains at rest and free from the action of any force save that inherent in its own molecules.

The delicate thermometer is placed in the test tube and lowered until the bulb is just above the disk. In order to secure an even temperature in all parts of the alcohol mixture in the vicinity of the disk the thermometer is moved from time to time in a circularly, pendulous manner. A tube prepared in this way will be suitable for use for several days, in fact, until the air bubbles begin to attach themselves to the disk of fat. In no case did the two liquids become so thoroughly mixed as to lose the property of holding the disk at a fixed point, even when they were kept for several weeks.

In practice, owing to the absorption of air, I have found it necessary to prepare new solutions every third or fourth day.

The disk having been placed in position, the water in the beaker glass is slowly heated and kept constantly stirred by means of the blowing apparatus already described.

When the temperature of the alcohol-water mixture rises to about 6 degrees below the melting point, the disk of fat begins to shrivel, and gradually rolls up into an irregular mass.

The thermometer is now lowered until the fat particle is even with the center of the bulb. The bulb of the thermometer should be small, so as to indicate only the temperature of the mixture near the fat. A gentle rotary movement should be given to the thermometer bulb, and I have thought it would be convenient to do this with a kind of clockwork, although I have not carried this idea into execution. The rise of temperature should be so regulated that the last 2 degrees of increment require about ten minutes. The mass of fat gradually approaches the form of a sphere, and when it is sensibly so the reading of the thermometer is to be made. As soon as the temperature is taken, the test tube is removed from the bath and placed again in the cooler. A second tube, containing alcohol and water, is at once placed in the bath. It is not necessary to cool the water in the bath. The test tube (I use ice water as a cooler) is of low enough temperature to cool the bath sufficiently. After the first determination, which should be only a trial, the temperature of the bath should be so regulated as to reach a maximum about 1°.5 above the melting point of the fat under examination.

Working thus with two tubes about three determinations can be made in an hour.

After the test tube has been cooled the globule of fat is removed with a small spoon attached to a wire before another disk of fat is put in.

Agreement of multiple determinations.

FILTERED BUTTER FAT.

	Degrees C.
No. 1, by one observer	33.5
No. 2, by another	33.5
No. 3, by a third	33.9
No. 4, by a third	32.4
No. 5, by a third	34.4

A second set of observations made with the same butter gave—

	Degrees C.
No. 1	33.7
No. 2	33.8
No. 3	33.5
No. 4	33.5

A different butter gave the following numbers:

	Degrees C.
No. 1	34.0
No. 2	33.7
No. 3	33.8
No. 4	34.0

Another butter, "Creamery Tub," gave the numbers below:

	Degrees C.
No. 1	33.7
No. 2	33.7
No. 3	33.6
No. 4	33.6

A neutral lard, from Armour & Co., Chicago, gave the following results:

	Degrees C.
No. 1	42.8
No. 2	42.4
No. 3	42.3
No. 4	42.6
No. 5	42.2
No. 6	42.0

An oleo oil, from Armour & Co., gave—

	Degrees C.
No. 1	29.4
No. 2	29.5
No. 3	29.5
No. 4	29.7
No. 5	30.0
No. 6	30.3
No. 7	29.7
No. 8	29.8

Another butter, shown by the microscope to be adulterated, gave—

	Degrees C.
No. 1	33.5
No. 2	33.7
No. 3	33.5

No. 1777, a doubtful butter gave—

	Degrees C.
No. 1	34.3
No. 2	34.5
No. 3	33.6
No. 4	34.0

No. 1779, also a doubtful butter, gave—

	Degrees C.
No. 1	34.2
No. 2	33.5
No. 3	33.0

These results show that the method is capable of general application.

Collecting together the mean results obtained with butter-fats the following table is obtained:

TABLE No. 1.—*Melting points, etc., of genuine butter.*

Serial number.	Melting point.	Per cent. soluble acid.	Specific gravity at 40° C.
	° C.		
1745	34.5	5.48	.911
1766	34.3	4.52	.910
1768	34.2	5.21	.910
1769	33.7	5.05	.912
1772	34.0	5.26	.911
1785	32.0	4.48	.912
1786	34.7	4.32	.912
Mean	33.8	4.86	.911

TABLE No. 2.—*Melting points, &c., of butters of doubtful purity.*

Serial number.	Melting point.	Per cent. Soluble acid.	Specific gravity at 40° C.
	° C.		
1777	34.1	3.02	.910
1770	33.6	3.16	.909
1780	34.4	3.02	.919
1781	34.5	3.97	.910
Mean	34.1	3.51	.909

The above were all bought as pure butters. They are condemned on account of the low percentage of soluble acid, while by their specific gravity they appear to fall near the limit of purity The soluble acid in the above was determined by washing out and not by Reichert's method.

TABLE No. 3.—*Melting point of substances sold as butter, but proved by analysis to be adulterated.*

Serial number.	Melting point.	Per cent. soluble acid.	Specific gravity at 40° C.
	° C.		
1755	39.0	1.53	.906
1778	33.6	0.21	.904
1787	34.6	0.09	.906
4594	35.3	0.09	.904
4595	37.8	0.90	.905

TABLE No. 4.—*Melting point, &c., of "oleo-oil" and "neutral lard" used as butter adulterants.*

Serial number.	Melting point.	Per cent. soluble acid.	Specific gravity at 40° C.
"Neutral lard":	° C.		
1754	42.4	0.10	.904
4597	42.4	....	....
"Oleo-oil:"			
1756	29.7	0.08	.903
4596	29.6	0.08	.903

TABLE No. 5.—*Melting point of mixtures made in laboratory as indicated.*

[The butter used had a melting point of 33°.1 C.; the "oleo-oil" of 29°.6 C.; and the "neutral lard" of 42°.4 C.]

No.	Composition of mixture.	Theoretical melting point.	Observed melting point.
		° C.	° C.
1	2 parts butter, 1 part "neutral"	36.2	35.2
2	1 part butter, 2 parts "neutral"	39.3	39.6
3	1 part butter, 1 part "neutral" and 1 part "oleo"	35.0	35.5
4	2 parts butter, 1 part "oleo"	31.9	32.0
5	1 part butter, 2 parts "oleo"	30.8	30.5

From the above it appears that the melting point of a mixture of two or more fats can be readily and accurately calculated from that of its constituents. The agreement, except in No. 1, is within the error of ordinary observation.

Remarks on preceding data.—The mean melting point of the butters examined is 33°.8 C., the maximum is 34°.7 C., and the minimum 32°.6 C. In general terms it may be said that a genuine butter will show a melting point falling within the limits of 33° and 34° C. Of butter adulterants the "neutral lard" has a comparatively high melting point and "oleo-oil" a low one. Unfortunately for analytical purposes it is easy for the fabricator to make an artificial butter whose melting point is sensibly the same as that of the genuine article. On the other hand it is seen that if a false butter be made of a genuine one and only one of the adulterants in common use, the variation of the melting point from the normal will be sufficiently great to call attention to the falsification.

EFFECT OF TIME ON MELTING POINT OF THE FAT DISKS.

By some variations in the melting point of fat disks of different ages my attention was directed to an investigation of the effect of time.

The following data will serve to measure the influence of age on the melting point:

BUTTERS.

Number.	Melting point.		Increase +; decrease—.
	Directly disk was made.	After 24 hours.	
	° C.	° C.	° C.
1	33.1	33.6	+0.5
2	34.3	34.7	+0.4
3	34.2	34.6	+0.4
4	34.5	35.2	+0.7
5	32.9	32.8	—0.1
6	33.1	34.5	+1.4

In every case except No. 5 in the above table it is seen that the melting point of the disks of butter was raised by standing on water at ordinary temperatures for twenty-four hours.

In one instance, a butter whose melting point was 34°.5 C. stood in the form of disks from May 27 until August 3. An attempt was made on this latter date to determine its melting point. At a temperature of 75° C. the disk had not assumed a spherical shape, and the temperature could be carried no higher on account of approaching the boiling point of the alcohol.

ADULTERATED BUTTERS.

Number.	Melting point directly disk was made.	Melting point after—	Increase.
	° C.	° C.	° C.
1	34.6	5 days 61.4	26.8
2	32.9	18 hours .. 37.0	4.1
3	38.1	24 hours .. 42.9	5.9
4	35.3	46 hours ...35.3	0.0
5	37.8	44 hours ...40.3	2.5

Again in every case but one a marked rise in the melting point.

"OLEO OIL."

	Melting point.
	° C.
"Oleo," at once	29.6
"Oleo," after 42 hours	42.5
Increase.	2.9

It would appear from the above results that adulterated butters and butter adulterants show a greater rise in melting points when the disks are a day or more old than pure butter. The analytical data, however, are too meager to permit a definite statement of this kind. Should it prove to be true, it would be a valuable indication in the discrimation between pure and adulterated butters. An examination of the old disks with the microscope did not reveal a crystalline structure, and this change, therefore, must be attributed to a molecular modification or superficial oxidation.

EFFECT OF THE PRELIMINARY HEATING OF THE FAT TO DIFFERENT TEMPERATURES.

A butter fat was melted at a low temperature and allowed to stand until the temperature had fallen to 30° C.; it was still perfectly fluid. The disks were formed by dropping on ice as usual. The melting point obtained was 33° C. The fat was now heated to 50° C. and treated as above; melting point, 33°.4 C. The temperature was then raised to 80° C.; melting point, 32°.8 C.

The above results, falling within the possible error of observation, show that the temperature to which the fat is subjected before the formation of the disks has no appreciable effect on the point at which the fat particle becomes a sphere.

EFFECT OF SUDDEN RISE OF TEMPERATURE.

A sudden rise of temperature tends to greatly lower the melting point. A fat which showed a melting point of 35°.3 C. when determined in the usual way, melted at once into a perfect sphere when dropped into the water-alcohol mixture having a temperature of 29° C. At 28°.5 C. the globule was irregular.

A disk of neutral lard, having by the usual method a melting point of 42°.4 C., became at once a sphere when dropped into the water-alcohol at 36°.2 C. Below that temperature the spheroidal shape was not symmetrical.

In all cases this phenomenon will appear. It may be suggested, therefore, with strict propriety, whether this may not be regarded as the proper melting point. Since the temperature at which the spheroidal state is assumed can be determined within one or two degrees by a preliminary trial, it would not be difficult to have a series of mixtures of water and alcohol arranged so as to show differences of temperature of 0°.5 C. By dropping the disks successively into these mixtures the instantaneous fusing point could be determined with accuracy.

The method set forth in the preceding pages has been proved by 165 determinations to be capable of giving agreeing results. Not only will the numbers obtained by the same observer be concordant, but also those of different analysts. This arises from the fact that the moment of the assumption of the spheroidal state is easily determined even by an unpracticed eye. I have also noticed that in this condition pure butter and oleo are quite transparent, while on the other hand neutral rd and adulterated butters are still somewhat opalescent. From this it is seen that the data obtained by the old method of determining the temperature of transparency would differ somewhat from those obtained by the proposed procedure. Since the age of the disk has a great deal to do with its melting point, I suggest that all determinations be made within fifteen minutes to two hours from the making of the disks.

The method can also be extended to such bodies as paraffine and bees-wax. The melting point of a paraffine was found to be—

	Melting point.
	° C.
No. 1	55.6
No. 2	55.1
No. 3	55.2
No. 4	55.3

An interesting phenomenon was observed in determining the melting point of the paraffine, which may be made to show, in a lecture experiment, the change of volume which bodies sometimes undergo in passing from a solid to a liquid state. The same mixture of water and alcohol used in the examination of fats, allowed the disk of paraffine to sink to about the same point as the disk of fat. When the temperature rose, however, to within one or two degrees of the melting point, there was a sudden increase in volume. The pellet of paraffine rapidly rose to the top of the tube. To avoid this and keep the globule within the liquid I made a mixture of water-alcohol and absolute alcohol. With this arrangement the rise of the paraffine was arrested in the upper third of the tube occupied by the absolute alcohol, where its assumption of the spheroidal state could be readily observed. On placing the tube in a cooling bath the globule of paraffine rapidly sinks as it solidifies. The disks of paraffine and bees-wax are quite irregular, but nevertheless suitable for the process. The melting point of the one sample of bees-wax examined was found to be 64°.2 C.

VISCOSITY.

The speed with which at identical temperatures and pressures different oils flow through an orifice may be used to dintinguish them from each other. For a description of the methods used in viscosimetry I refer to Allen's Com. Organic Analysis.[1] An ingenious and useful apparatus for viscosimetry has been invented by Babcock.[2]

Babcock has applied his apparatus to the investigation of the viscosity of butter soaps with promising results.[3]

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[1] Vol. 2, 2d ed., pp. 194 *et seq.*

[2] Fifth Ann. Rept. Bd. Control N. Y. Exp. Sta., pp. 316 *et seq.*

[3] *Ibid.*, pp. 338 *et seq.*

REFRACTIVE INDEX OF OILS.

The use of the refractometer of Abbé in the examination of butters has been proposed by Müller.[1] The principle of the use of this instrument is, that the fats of pure butter possess a less refractive power than the glycerides of a higher molecular weight.

This subject has also been treated by Skalweit.[2]

ESTIMATION OF SOLUBLE ACIDS IN BUTTER FATS.

Method of Hehner and Angell.[3]—Hehner and Angell, in June, 1874, published a pamphlet on butter analysis in which the details of their method were given.

The following is an abstract of this method:[4]

A weighed quantity, usually 3 grams, of the fat was saponified in a porcelain dish with caustic potash, with frequent stirrings with a glass rod. The clear butter soap was transferred to a flask or retort and decomposed by means of dilute sulphuric acid. This mixture, which contained sulphate of potash, glycerine, and the volatile acids in solution and the insoluble fatty acids floating on the top, was distilled, and the acidity of the distillate estimated by means of a soda solution of known strength. The practical difficulties of this method, such as the violent bumping of the boiling liquid and the impossibility of obtaining a distillate perfectly free from acid, led the authors to adopt a somewhat different method.

This modification is based upon the different percentages of the insoluble fatty acids in butter and other animal fats. The insoluble acids, after saponification, were collected on a moistened filter paper, washed with hot water, and when the soluble acid was washed out, dried and weighed.

They found the percentage of insoluble fats in butter to vary from 85.40 to 86.20, while in other animal fats the percentage of insoluble fatty acids was about 95.5. As will be shown further along, a small error is introduced into this method by washing the insoluble fatty acids on the filter. When this error is avoided, it is found that the percentage of the insoluble fatty acids in butter fat is considerably higher than the figure which has just been given. A detailed description of this part of the process will be given farther on. Turner[5] suggested the employment of alcohol, with the view to hasten the saponification of the fat; a modification of the process which has been almost universally adopted by analysts.

About 30 or 40cc. of spirits of wine are added to the butter in the porcelain dish and heated over the water bath to near the boiling-point.

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[1] Archiv d. Pharm., 1886, p. 210.

[2] Rep. d. Ver. Anal., Chem., 1886, p. 181.

[3] Analyst, 1877, p. 147.

[4] Hassall, Food and its adulterations, p. 446.

[5] *Ibid.*, p. 447.

About 5 grams of solid caustic potash are then added, and from time to time a few drops of water, to facilitate its solution, the liquid being stirred all the time. In this manner the butter becomes rapidly saponified. The clear yellowish solution is then freed from all alcohol over the water bath and the soap decomposed as already described. Care should be taken to remove all the alcohol, as a small quantity of the fatty acids might be held dissolved should any alcohol remain, and so lead to an erroneous result.

Hehner's method modified by Reichert.[1]—Weigh out 2½ grams of dried and filtered butter fat in an Erlenmeyer flask of 150cc. capacity; add 1 gram of solid potassium hydrate and 20cc. of 80 per cent. alcohol. This mixture is kept upon the water bath with constant shaking until the soap obtained no longer forms a foamy, greasy mass. Afterwards 50cc. of water are added to the flask, and the soap, after it has dissolved in water, is decomposed with 20cc. of dilute sulphuric acid (1cc. of pure sulphuric acid to 10cc. of water). The contents of the flask are now subjected to distillation, with the precaution of conducting through it a slow stream of air, in order to avoid bumping. It is also recommended to use a bulb tube with a wide opening, in order to avoid carrying over the sulphuric acid. The distillate, which, especially with fats poor in butter and by rapid distillation, always deposits a little of the solid fat acids, is filtered through a moistened filter paper and collected in a 50cc. flask. After 10 to 20cc. are passed over it is poured back into the flask and the distillation is now continued until the distillate amounts to exactly 50cc The distillate, which, when the distillation has gone on evenly, forms a water clear liquid, is immediately titrated with decinormal soda lye after the addition of 4 drops of litmus tincture. The titration is finished if the blue color of the litmus remains constant for some time. Six analyses of an artificial butter fat required 10.5cc. of decinormal soda lye to neutralize the acid in the distillate.

The genuine butter gave on three trials 14 50, 14.45, and 14.60cc., respectively, of the decinormal soda.

Two samples of cocoanut fat required 5.70 and 3.70cc. of soda lye.

Thirteen samples of pure butter required a mean of 13.97cc. of the decinormal soda.

All the other fats which are used in the adulteration of butter required a much smaller amount of the decinormal soda for the saturation of the distilled acid.

In artificial butters the proportion of pure butter and added fat may be calculated from the following formula:

$$B = a\,(n - b).$$

in which n represents the most probable value of the number representing the quantity of decinormal soda solution required either for pure butter or for the fat with which it may be adulterated. When B equals

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[1] Zeit. Anal. Chem., 1879, pp. 68, *et seq.*

O, that is, when the substance contains no pure butter, the value of n may be taken at .30. We have, therefore,

$$O = a\ (0.30 - b) \text{ from which } b = .30.$$

When B is equal to 100, that is, when the butter is pure, as has already been said, the most probable value of n, according to the thirteen analyses given, is 13.97, or in round numbers 14 ± 45, then we have the equation

$$100 = a\ (14.00 \pm 0.45 - 0.30)$$

and from this the value of

$$a = 7.30 \pm .24.$$

The above equations may therefore be condensed into $B = (7.30 \pm 0.24)\ (n - 30)$, that is, in order to find the probable butter content of a fat mixture subtract from the number of the cubic centimeters of decinormal soda lye used for titration .30 and multiply the remainder by 7.30. The probable error which will be met with by this estimation amounts to $\pm 0.24\ (n - 0.30)$.

Medicus and Scherer[1] examined the method of Reichert and found it to be quite exact. For pure butter they found the quantity of decinormal soda lye required should be 13cc.; a mixture of equal parts of butter fat and tallow required 7cc.

Two parts of butter fat and one of tallow required 9.1cc.; three parts of butter fat and one of tallow required 10.1cc.

The authors call attention to the fact that melted butter fat slowly cooled may separate into portions requiring different quantities of the decinormal soda for the saturation of the distilled acid which they afford. Two and one-half to 3 pounds of pure butter fat were used. This was melted and allowed to cool with continued stirring in order to secure a perfectly homogeneous mass. 2½ grams of this mixture, by Reichert's method, required 14cc. of decinormal soda. The fat was now again melted, poured into a large beaker glass, and uncovered allowed to cool without stirring. The solidification took place slowly. After solidification 2½ grams from the upper layer required 13.3cc. of soda.

Allen[2] also highly recommends Reichert's method. He uses it as follows: Weigh out ~~25~~ grams of the clarified butter fat and saponify in a closed flask (a closed flask has been used in the work of the Chemical Division with butter since 1883) with 25cc. of approximately $\frac{N}{2}$ KOH. Transfer the product to a porcelain basin and evaporate the alcohol at a steam heat. Dissolve the residue in water, add some fragments of pomace coiled round with platinum wire, and distil gently until 50cc. have passed over. Titrate the distillate with $\frac{N}{10}$ caustic alkali using phenol-phthalein as an indicator.

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[1] Zeit. Anal. Chem., 1880, pp. 159 *et seq.*

[2] Analyst, 1885, p. 103, *et seq.*

Allen found that a genuine butter fat required not less than 12.5cc. $\frac{N}{10}$ alkali for neutralization of the acid in distillate and that this corresponds to 3.9 per cent. butyric acid distilled over, so that somewhat over 4 per cent. of volatile acids in terms of butyric may be considered to be yielded by the process. (Instead of 3.9 per cent. it should be 4.4 per cent. since 1cc. $\frac{N}{10}$ caustic alkali neutralizes .0088 grams butyric acid.)

Allen gives some comparative results with Reichert's method obtained by different chemists. In the conclusion of the paper Reichert's method is said to be more enlightening than Koettstorfer's for sorting butters.

Modification of Reichert's method by Dr. B. F. Davenport (communicated in MSS.)—Use only 10cc. of alcohol in the saponification; the advantage being that with this small quantity the saponification is almost immediate. In fact I begin to draw out the alcoholic vapor from the flask as soon as it comes to the boil, using a water pump; thus it takes only about fifteen minutes to complete the saponification and the mass evaporated down to a thick mass, free from any alcoholic vapors. My process is to melt the butter at about 80° C.; filter off the clean fat, stir it into a uniform mass as it solidifies. Weigh off two portions (for I work in duplicate) of the solid mass of 5 grams each upon counterpoised double filters of about the size of the scale pan, using double filters that there may be no chance of anything going through to the scale pan. Roll up the edges of the double filter over the butter upon them, and slip it all into an Erlenmeyer flask. It is easy to get the exact 5 grams upon the open filter, and by putting all into the flask there can be no loss in the transfer. Run off upon the butter in the flask 10cc. of 70 per cent. alcoholic solution, containing 2 grams of KOH. Saponify and get dry mass in about fifteen minutes, add to it 100cc. of water, dissolve, aided by heat, add 50cc. of dilute H_2SO_4 containing about one-tenth part of commercial H_2SO_4, add several pieces of rough pomace loaded with enough stout platinum wire to lie upon the bottom of the flask, and then distill off 100cc. directly into a sugar flask having a small funnel, and filter in its mouth.

I used at first after distilling off about 20cc. to pour it back into the flask and then distill off 100cc., but I soon learned that that made no difference, as also using a 50cc. sugar flask full of diluted H_2SO_4, instead of the directed 40cc., which was not quite so convenient a quantity to measure off. I leave the end of the bulb tube connecting the flask with the condenser long enough to enter the condenser so far that there is no need of any rubber connection between them, there being no escaping at the upper end of the condenser of any vapor or of scarcely any odor at all. The operation requires no transferring of the material from the beginning to end. Samples of known pure butter have by this method required an average of 28.8cc. of alkali to neutralize the 100cc.

of distillate. I use phenol-phthalein as an indicator instead of litmus. When the alcoholic KOH solution has been made for some time I make a blank saponification and distillation with that and discount the cubic centimeters of soda solution required by that for that required by the butters.

Reichert's method has also been tried and approved by Caldwell.[1] He says:

> For foreign fats Reichert's method was followed with much satisfaction. When all the necessary solutions are once prepared the analysis is made with comparatively little trouble; with less, in fact, than is allowed even by those who praise it most. Its author says that a current of air must be passed through the liquid in the flask while the distillation is going in, to prevent bumping, and Ambuhl says that all attempts to dispense with this precaution by the use of pumice-stone, platinum scraps, and the like failed. Nevertheless, finding it very inconvenient to use the current of air, I ventured to try a combination of short spirals of platinum wire and pieces of pumice stone together, and with complete success; the ebullition continued from beginning to end as quietly as could be desired.

Meissel[2] has described a modification of Reichert's process as follows:

Five grams of the melted and filtered butter fat are treated in a 200cc. flask with 2 grams of stick alkali and 50cc. 70 per cent. alcohol. After complete saponification the alcohol is evaporated. The soap is dissolved in 100cc. water and decomposed with 40cc. one-tenth H_2SO_4. The flask is supplied with some pieces of pumice-stone and connected by means of a bulb with a condenser.

The distillation is continued until 110cc. are drawn over. After filtration 100cc. are titrated in presence of litmus with one-tenth N potash and the number of cubic centimeters required increased by one-tenth.

If less than 26cc. of the alkali solution are required in the titration the butter may be suspected of falsification.

Mode of procedure in Reichert's method (used by Dr. C. A. Crampton, Department of Agriculture).—About 2.5 grams of the melted butter fat are weighed out by means of a small pipette and beaker, which are weighed again after the sample has been taken out, and run into a bottle provided with a patent india rubber stopper; 25cc. of a solution of (approximately) semi-normal alcholic potash is added, the bottle closed and placed in the steam bath until the contents are entirely saponified, facilitating the operation by occasional agitation. The bottle is then removed from the bath, allowed to stand a few moments until partially cooled off, when its contents are transferred to a porcelain evaporating dish, the bottle being rinsed with a little alcohol. The alcohol is then driven off as rapidly as possible, and when the mass of soap and alkali is nearly dry, it is dissolved up in 25cc. of water, and transferred to a suitable flask of about 200cc. capacity, which is fitted with a delivery tube and condenser; the delivery tube is carried up about 8 inches before it is bent to enter the condenser and a bulb is blown in it just below

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[1] Second Ann. Rept. N. Y. S. Bd. of Health, p. 526.

[2] Ding. Poly. J., vol. 233, p. 229.

the elbow and filled with broken glass or glass wool. After the soap solution has been transferred to this flask, the evaporating dish is rinsed out with 25cc. more water, which is added to the contents of the flask, and the fatty acids are then set free by the addition of 20cc. of a solution of phosphoric acid,[1] making the liquid measure in all about 70cc. Heat is applied gently at first, and gradually increased until the distillate comes over regularly. When 50cc. have distilled off the operation is finished and the distillate is titrated with one-tenth alkali, using phenolphthalein as an indicator.

I have adopted phosphoric acid in preference to sulphuric for setting free the fatty acids, because it is not so liable to carry over as the latter; much greater care is necessary when sulphuric acid is used. Before the modification of the delivery tube was adopted, I frequently found H_2SO_4 in the distillate. Thus, before using the bulb, two blank experiments required 1.8 to 2.0cc., one-tenth alkali, for neutralization and gave a perceptible precipitate of $BaSO_4$. After adding the bulb I found blanks occasionally to require as much as .8cc. when the distillation had not been carefully watched. The following comparative results show that there is practically no difference which acid is used, when the operation is carried on with care. The processes used were identical, except that in the second, 20cc. of 10 per cent. sulphuric acid was substituted for the phosphoric acid. The results are for 2.5 grams of fat.

No. 1	With phosphoric acid	12.7	12.6	12.7		
	With sulphuric acid	12.7	12.7	12.6		
No. 2	With phosphoric acid	15 8				
	With sulphuric acid	15.3				
No. 3	With phosphoric acid	13.1				
	With sulphuric acid	13.2				
No. 4	With phosphoric acid	15.3	14.1	14.5		
	With sulphuric acid	14.8	14.6	14.9	14 5	15.0

Blanks should always be run, and will be generally found to require .1 to .3cc. of the deci-normal soda before they will show the color with the phenol indicator.

Koettstorfer's process[2] (*as used in this laboratory*).—About 2.5 grams butter fat (filtered and free from water) are weighed into a patent rubber-stoppered bottle and ~~25~~cc. (approximately) semi-normal alcoholic potash added. 5 The exact amount taken is determined by weighing a small pipette with the beaker of fat, running the fat into the bottle from the pipette and weighing beaker and pipette again. The alcoholic potash is measured always in the same pipette and uniformity further insured by always allowing it to drain the same length of time (thirty seconds). The bottle is then placed in the steam bath together with a blank, containing no fat. After saponification is complete, and the bottles cooled down, the contents are titrated with accurately semi-normal hydro-

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[1] Made by dissolving 200 grams of commercial glacial phosphoric acid in a litre of water; its specific gravity is 1.140.

[2] Zeit. Anal. Chem. 1879, p. 199; Analyst, 1879, p. 106.

chloric acid, using phenol-phthalein as an indicator. The number of cubic centimeters of the acid used for the sample deducted from the number required for the blank gives the number of cubic centimeters which combines with the fat, and the saturation equivalent is calculated by the following formula, in which W equals the weight of fat taken in milligrams and N the number of cubic centimeters which has combined with the fat.

$$\text{Sat. Equiv.} = \frac{2\,W}{N}.$$

For pure butters the mean value of N is about ~~17~~ when 2.5 grams of butter fat are taken, and the saturation equivalent may vary from 230 to 255. On the other hand for lards, tallows, and other fats commonly used for adulterants the equivalent rises to 270 and 290. These numbers, therefore, give a fair idea of the purity of a butter, or if an adulteration has been practiced, of its extent.

ESTIMATION OF INSOLUBLE ACIDS IN BUTTER FAT.

Method of Hehner.[1]—This method consists in saponifying the fat with alcoholic caustic potash, subsequent evaporation of the alcohol, decomposition of the soap with sulphuric or hydrochloric acid, and the determination of the insoluble acid gravimetrically.

The process as originally described by Hehner is carried on as follows:

The filtered butter fat is weighed in a beaker glass with a glass rod; 3 or 4 grams are taken out by means of the glass rod and put in an evaporating dish about 5 inches in diameter; the glass rod with the fat which remains on it is left in the evaporating dish. The beaker glass is again weighed and the amount of butter fat determined from the difference in weight. To the weighed fat are added 50cc. alcohol and 1 to 2 grams of pure caustic potash. The alcohol is warmed gradually upon the water bath, by which the butter fat, especially when stirred with the glass rod, easily dissolves to a clear yellow liquid, giving off a distinct odor of butyric ether. The heating is continued for about five minutes and distilled water is then added drop by drop to the mass. If this produces a cloudiness in the liquid, due to the separation of undecomposed fat, the heating is continued somewhat longer until finally the further addition of water does not produce the least cloudiness. Should, however, through the careless addition of water, some fat separate in the form of oily drops which do not again easily pass into the solution in the diluted alcohol, the whole mass must be evaporated to dryness and treated anew with alcohol, or the experiment be done over again with some fresh fat.

The clear soap solution is now evaporated on the water bath to the consistency of sirup in order to remove the alcohol, and the residue

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[1] Zeit. Anal. Chem. 1877, pp. 145 *et seq.*

dissolved in 100 to 150cc. of water. To the clear liquid hydrochloric or sulphuric acid is added to a strongly acid reaction, in order to decompose the soap. The insoluble fat acids are now separated out as a cheesy mass, which for the most part quickly rise to the surface. The heating is continued for a half hour until the fat acids are melted to a clear oil and the acid aqueous liquid is almost completely clear. Meanwhile a thick Swedish filter paper of 4 or 5 inches in diameter has been dried in a water bath. The filter paper must be of the best quality and so thick that even hot water will only pass through it drop by drop. A small beaker glass is now weighed, afterwards a filter tube, and then the filter tube and the filter; in this way is obtained the weight of the filter and the beaker glass.

The weighed filter is now fitted to a funnel moistened and half filled with water. The aqueous liquid and the melted fat are then poured out of the evaporating-dish into the filter, and the dish and glass rod are washed with boiling water. There is no difficulty in bringing all of the fat on the filter, so that the evaporating dish does not appear in the least greasy. To make sure, however, the dish can be washed with ether and the liquid obtained added to the fatty acids.

The fatty acids are washed upon the filter with boiling water. The filter should be never more than two-thirds full. If the filtrate tested with sensitive litmus tincture does not appear acid, the rest of the water is allowed to run through, and the funnel is dipped into a beaker-glass filled with cold water, so that the surface of the liquids within and without the funnel are at the same level. As soon as the fatty acids have solidified the filter is taken out of the funnel, placed in the weighed beaker-glass, and dried in a water-bath to constant weight. The drying is continued for two hours and the filter paper is then weighed. It is again dried for two and a half hours and weighed a second time. It must be remembered that it is not a mineral substance which is under treatment, but an easily-oxidizable fat, so that an exact constant weight cannot be expected.

Butter fat gives between 86½ and 87½ per cent. of insoluble fatty acids, though in some cases the number may rise to 88 per cent. On the other hand, the animal fats give about 95½ per cent. of insoluble fats.

It must be expected that the kind of food which cows receive influence considerably these numbers. In order to determine this important point, Dr. Turner had a cow fed for a long time exclusively on oil cake, with the object of raising the percentage of insoluble fat acids to the highest point. It is worthy of remark, however, that the butter so produced gave the unusually low percentage of 86.3.

Method of Muter.[1]—The total fatty acids. About 10 grams (or 150 grains) of the butter fat at 100° F. are weighed by difference from a suspended tube into a clean, dry 15-ounce flask, and 5 grams of potassium hydrate with 2 fluid ounces of rectified spirit are added. The flask is

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[1] Analyst, 1877, pp. 10, 11.

placed in a basin with hot water, and kept boiling for a considerable time, until on adding water not the faintest turbidity occurs. Ten ounces of water are added, the evaporation continued (just short of boiling) until all traces of alcohol are dissipated. The contents of the flask are then made up to 7 ounces with nearly boiling water, and a good-fitting cork having been introduced through which just passes a tube 2 feet long and ending in a small funnel, 5 grams of full strength sulphuric acid are poured in down the tube followed by some water. The whole is then agitated with a circular motion until the soap, which rises suddenly, is changed into a perfectly clear and transparent stratum of fatty acids. The flask and contents are then cooled down to 40° F., till a perfectly solid cake of fatty acid forms. A few drops of cold water are run in to wash the tube, and, the cork having been removed, a small piece of fine cambric is placed over the mouth of the flask, held *in situ* by an ordinary India-rubber ring. The fat cake is caused to detach itself from the sides of the flask by a gentle movement, and then the filtrate is decanted, without breaking the cake, into a litre test mixer with a good stopper. About an ounce of cold water is poured into the flask through the cambric, and the whole cake and flask rinsed out by gently turning round, and the washings added to the filtrate. Six ounces of water at 120° F. are now added through the muslin, which is then quickly detached, and the cork and tube inserted; the whole again heated, this time to 200° F., and kept constantly agitated with a circular but not a jerky motion for five minutes. This agitation so divides the fat that it almost forms an emulsion with the water, and is the only means of thoroughly and rapidly washing fatty acids without loss. In practice no butyric acid comes off at 200° F., but any trace that might do so is caught in the long tube. The cooling and filtering are then again proceeded with as above described (the filtrate being added to the contents of the test mixer), and the washings are repeated alternately, cold with 1 ounce, and hot with 6 ounces of water, until they do not give the slightest change to neutral litmus. After thoroughly draining the residual cake by letting the flasks stand upside down for some time, the cambric is removed and the flask is laid on its side in the drying oven, with a support under the neck, until the acids are thoroughly fused, when they are poured while hot into a tarred platinum capsule, dried and weighed. The film of fatty acid still remaining on the flask is rinsed out with ether and dried in a small weighed beaker, and the weight added to the whole. If any drops of water be observed under the fatty acids in the capsule after an hour's drying the addition of a few drops of absolute alcohol will quickly cause them to dry off. If any trace of fat is on the cambric it should be also dried and extracted with ether, but with care not to break the cake at the last pouring off this does not occur.

The process is absolutely accurate, and the merest tyro cannot make any loss so long as he does not deliberately shake the melted acids against the cork, which he could not do if he practiced a circular agitation while washing.

The filtrate in the test mixer is now made to a definite bulk of 1 litre, and in 200cc. the total acidity is taken with a weak solution of sodium hydrate. The solution I generally use represents .01 of NH_3 in each cubic centimeter, as it serves also for nitrogen combustions; but a useful strength would be decinormal soda, containing .004 NaOH in each cubic centimeter. The acidity found is multiplied by 5, calculated to $H_2\,SO_4$ and noted as "total acidity as H_2SO_4"; 100cc. are next taken, and precipitated with barium chloride in the presence of a strong acidulation, with hydrochloric acid, well boiled and washed by three decantations, boiling each time; and lastly on a filter, till every trace of soluble barium is removed. The precipitate is dried, ignited, and weighed as usual, multiplied by 10, and calculated to H_2SO_4 and noted as "total sulphuric acid." Lastly, 100cc. are evaporated to dryness over the water bath in a tarred platinum dish holding 120cc. and furnished with a cover of platinum foil, also tarred. When dry the dish is covered and heated over a Bunsen till all fumes cease, and, a fragment of pure ammonium carbonate having been added, the whole is again ignited and weighed. The amount of potassium sulphate found is multiplied by 10 and calculated to H_2SO_4 and noted as "combined sulphuric acid."

OTHER METHODS.

Liebschütz[1] has described a method for the examination of butter and oleomargarine, being a modification of David's process.[2]

The fatty acids are saponified by baryta in alcoholic solution. The alcohol is evaporated and the glycerine washed out. The excess of baryta is removed by exactly neutralizing with sulphuric acid and filtering. The residue, however, is not merely a mixture of glycerine and water. The addition of alcohol in excess throws down a considerable quantity of salts which have remained in solution. The alcohol is again evaporated and the glycerine obtained, dried, and weighed. Pure butter yields about 13.7 per cent. of glycerine in this way, while oleomargarine yields only 7 per cent. The glycerine from butter when ignited left about 5 per cent. ash (barium) while that from oleo left only .3 to .6 per cent.

RESULTS OF HANSSEN'S INVESTIGATIONS.

Dr. August Hanssen[3] has made a comparative study of the more important methods of analysis mentioned in the foregoing pages and has reached the following conclusions:

(1) The determination of the melting-points of the different fats is to be strongly recommended.

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[1] Analyst, 1885, p. 111, *et seq.*

[2] Compt. Rend., 1886, vol. XCIV, p. 1427.

[3] Studien über den chemischen Nachweis fremder Fette im Butterfette, p. 34.

(2) The elementary analysis of the fats gives no indication whether adulteration has been practiced or not.

(3) Butter fat is not easily decomposed by heat. With a rise of temperature the decomposition is at first, for the greater part, confined to the glycerides of the non-volatile acids.

(4) In the saponification of butter fat by Hehner's method there is no appreciable loss of ethers. There is also no loss of volatile acids in direct saponification in alcohol.

(5) For the detection of foreign fats in butter, the best method is that of Reichert-Meissel, and next that of Koettstorfer.

(6) For a comparative test of the various methods the mean for insoluble acid (Hehner) is taken at 87.50 per cent.; for Koettstorfer's equivalent 227, and for Reichert-Meissel 28.8.

(7) The washing out of the soluble acids must not be carried too far; for 2 to 2.5 grams of fat three litres of water seem best.

ABSORPTION OF BROMINE AND IODINE BY BUTTER FATS.

Oleic acid is capable of absorbing for each formula molecule one molecule of bromine or iodine. Stearic acid does not possess this property. Therefore it is easy to approximately determine the relative quantities of these two acids when present in the same fat by the quantity of the halogen absorbed.

Thus (stearic acid) $C_{18}H_{36}O_2$ does not absorb bromine and iodine, while (oleic acid) does.

$$C_{18}H_{34}O_2 + \frac{I_2}{Br_2} = C_{18}\,H_{34}\frac{I_2}{Br_2}O_2.$$

The glycerides of the above acids, *i. e.*, the natural fats, have the same absorptive power as the acids themselves.

Mills, Snodgrass & Akitt[1] have determined the quantities of bromine absorbed by various fixed oils. The method employed is as follows:

The weight of dry oil taken is about .1 gram; this is dissolved in a stoppered bottle of 100cc. volume by 50cc. dry CCl_4. To this is now added a solution of about 8 grams per litre of bromine dissolved in CCl_4. The addition of this reagent is continued until a permanent coloration is produced at the end of fifteen minutes.

If greater accuracy is required an excess of bromine may be added, afterwards treated with a solution of KI and some starch, and titrated with a standard solution of sodium thiosulphate.

The excess of bromine may also be determined by titration with a standard solution of β-naphthol in CCl_4

Hübl[2] has described the reactions of fats with iodine.

The reagents employed are an alcoholic solution of iodine and $HgCl_2$, in the proportion I_2: $HgCl_2$.

The iodine is dissolved (25 grams) in absolute alcohol (500cc.) The mercuric chloride is also dissolved (30 grams) in nearly absolute alcohol

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[1] Journ. Soc. Chem. Industry, vol. 2, p. 435, and vol. 3, p. 366.

[2] Ding. Poly. J., vol. 243, p. 281.

(500cc.). After filtering it is added to the solution of iodine. After standing twelve hours its iodine strength is determined by titration with decinormal solutions of sodium thiosulphate. From .8 to 1 gram of the fat is dissolved in 10cc. chloroform. To this, in a stoppered bottle, is added the solution of iodo-mercuric chloride (20 to 30cc.) After standing for two hours the solution must still be brown.

Add now 10 to 15cc. 10 per cent. water solution of KI and dilute with water to 150cc. The free iodine is then determined by standard thiosulphate of sodium. The compound formed when pure oleic acid is treated as above is chloro-iodo-oleic acid ($C_{18}H_{34}IClO_2$).

Moore[1] has tried Hübl's method and finds it valuable.

The fat of butter containing less oleic-glycerides than the fats ordinarily used as adulterants for butter shows, consequently, less bromine or iodine absorption:

Kind of fat.	Absorption of bromine.	Absorption of iodine.
	Per cent.	*Per cent.*
Butter fat	24.5 to 27.9	26. to 35.
Lard..................	37.3	59. 61.9
Tallow	....	40.
Cotton-seed oil......	50.	105. 109.
Cocoanut oil	5.7	8.9

The method is therefore of value in determining the nature of the fat under examination.

If there be a mixture of two fats the methods will also give a fairly good approximation of the percentages of each.

Thus, let x be the percentage of one fat and y of the other.

Then—

$$x + y = 100$$

Let m be the representative of the iodine absorption of x and n of y, and let A be the number found for the mixture.

Then—

$$x = \frac{100\,(A - n)}{m - n}$$

Jones[2] points out the changes which butter fats undergo when kept for a long while at a high temperature. He notices in a few hours that the specific gravity of such a fat kept at 100° F. increased from 912.1 to 912.6. He uses the following method of estimating the insoluble fatty acids:

REAGENTS.

(*a*) Twenty-eight grams roughly weighed of the best potassium hydrate dissolved to a litre with alcohol, specific gravity .840.

(*b*) Twenty-five grams of strong sulphuric acid made up to a litre of distilled water.

(*c*) Decinormal soda solution of exact strength.

Saponification is carried on in flasks about 250cc. capacity. About 5 grams of butter fat are used for each saponification. The alcoholic

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[1] Am. Chem. Jour., vol. 6, p. 416.

[2] Analyst, 1878, pp. 19 *et seq.*

potash is measured by 50cc. pipette, which is allowed to drain into each flask for exactly the same length of time. The flasks are closed with glass marbles, placed upon the water bath and saponified at a temperature of about 50° C. After perfect solution has taken place they are allowed to remain for an hour or two and then diluted with slightly warmed distilled water. Into each flask and likewise into two beakers containing 50cc. of the alcoholic potash are now run about 1cc. of the approximately semi-normal acid more than is necessary to neutralize the 50cc. of alcoholic potash. The excess of the acid over the potash is afterwards determined by the decinormal soda. The flasks after the addition of the acid are nearly filled with water and gently agitated, then placed on the water bath until the fatty acids form a clear stratum. They are then allowed to cool and stand over night. On the following morning the solutions from the cakes of fat are poured into a filter. When the whole solution is on the filter the flasks are rinsed with 15 to 20cc. of cold distilled water, and when this is poured off about 150cc. of hot water are added and the flasks briskly shaken for a minute or two. Two good washings with hot water are believed to be enough. The filtrates are now treated with the decinormal soda, the amount for the excess of sulphuric acid deducted, the remainder being the index of the soluble acids of the butter, which are calculated as butyric acid.

The insoluble fatty acids in the flasks and the small amount that may have passed on to the filter paper are allowed to remain until the following day, by which time the latter become air-dried and in a fit state to rinse with ether. The fat in the flasks is then melted and poured, together with the rinsings of the ether, into counterpoised dishes with perpendicular sides, about 3 inches across and 1½ inches deep, and the filter papers are also thoroughly washed with ether, the funnels being covered during the process. After the evaporation of the ether a little absolute alcohol is added, the dishes dried in the water-bath for half an hour, cooled, and weighed. Afterwards they are again dried for twenty minutes and reweighed.

For a more convenient method of manipulating fatty acids, Blyth[1] has recommended the following:

The flask in which the saponification is made should be of 300 to 400cc. capacity, with a rather long and narrow neck, furnished with an accurately fitting stopper, through which two tubes pass, one provided with a stop-cock to let out the liquid, and therefore terminating on a level with the interior surface of the stopper, the other to let in the air, prolonged to nearly the bottom of the flask and externally bent siphon-like. The fat is saponified in the flask and the soap decomposed in the usual way; when this is effected, the stopper is inserted, and the flask is turned upside down and kept in that position during the entire washing process. Directly the whole of the fat has risen to the surface the lower liquid is run off, whilst hot or cold water is introduced by opening

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[1] Analyst, 1878, p. 112.

the stopper under the water and simultaneously sucking at the syphon. Thus all waiting for the fat to cool is discarded, and reasonable quantity of water can be rapidly used to thoroughly wash the fatty acids, and a filter is not required.

DETERMINATION OF SOLUBLE AND INSOLUBLE FAT ACIDS.

METHOD ADOPTED BY ALLEN.[1]

(*a*). Dissolve 14 grams of good stick-potash in 500cc. of rectified spirit, or methylated spirit which has been redistilled with caustic alkali, and allow the liquid to stand till clear. This solution will be approximately seminormal.

(*b*). A standard hydrochloric or sulphuric acid of approximately seminormal strength.

(*c*). Accurately prepared decinormal caustic soda. Each 1.0cc. contains .0040 grams of NaOH and neutralizes, .0088 grams of butyric acid, $C_4H_8O_2$.

A quantity of the butter fat (separated from water, curd, and salt, as described on page 152) is melted in a small beaker, a small glass rod introduced, and the whole allowed to cool, and then weighed. It is remelted, stirred thoroughly, and about 5 grams poured into a strong 6-ounce bottle. The exact weight of fat taken is ascertained by reweighing the beaker containing the residual fat.

By means of a fast-delivering pipette 50cc. measure of the alcoholic potash (solution *a*), is run into the bottle, and the pipette drained exactly thirty seconds. At the same time another quantity of 50cc. is measured off in an exactly similar manner into an empty flask.

The bottle is fitted with an india-rubber stopper, which is tightly wired down, and is placed in the water-oven, and from to time removed and agitated, avoiding contact between the liquid and the stopper. In about half an hour the liquid will appear perfectly homogeneous, and when this is the case the saponification is complete, and the bottle may be removed. When sufficiently cool, the stopper is removed, and the contents of the bottle rinsed with boiling water into a flask of about 250cc. capacity, which is placed over a steam bath, together with the flask containing merely alcoholic potash, until the alcohol has evaporated.

Into each of the two flasks is now run about 1cc. more seminormal acid (solution *b*) than is required to neutralize the potash, and the quantity used accurately noted. The flask containing the decomposed butter fat is nearly filled with boiling water, a cork with a long upright tube fitted to it, and the whole allowed to stand on the water-bath until the separated fatty acids form a clear stratum on the surface of the liquid. When this occurs the flask and contents are allowed to become perfectly cold.

Meanwhile the blank experiment is completed by carefully titrating the contents of the flask with the decinormal soda, a few drops of an alcoholic solution of phenolphthalein being added to indicate the point of neutrality.

The fatty acids having quite solidified, the resultant cake is detached by gently agitating the flask, so as to allow the liquid to be poured out, but avoiding fracture of the cake. The liquid is passed through a filter to catch any flakes of fatty acids, and is collected in a capacious flask. If any genuine butter be contained in the sample, the filtrate will have a marked odor of butyric acid, especially on warming.

Boiling water is next poured into the flask containing the fatty acids, a cork and long glass tube attached, and the liquid cautiously heated till it begins to boil, when the flask is removed and strongly agitated till the melted fatty acids form a sort of emulsion with the water. When the fatty acids have again separated as an oily layer, the contents of the flask should be thoroughly cooled, the cake of fatty acids detached,

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[1] Commercial Organic Analysis, vol. 2, 2d ed., pp. 156 *et seq.*

and the liquid filtered as before. This process of alternate washings in the flask by agitation with boiling water, followed by cooling, and filtration of the wash-water, is repeated three times, the washings being added to the first filtrate. It is often difficult or impossible to obtain the wash-water wholly free from acid reaction, but when the operation is judged to be complete the washings may be collected separately and titrated with decinormal soda. If the measure of this solution required for neutralization does not exceed 0.2cc. further washing of the fatty acids is unnecessary.

The mixed washings and filtrate are next made up to 1,000cc., or some other definite measure, and an aliquot part carefully titrated with decinormal soda (solution *c*). The volume required is calculated to the whole liquid. The number so obtained represents the measure of decinormal soda neutralized by the soluble fatty acids of the butter fat taken, plus that corresponding to the excess of standard acid used. This last will have been previously ascertained by the blank experiment. The amount of soda employed in this is deducted from the total amount required by the butter fat quantity, when the difference is the number of cubic centimeters of standard soda corresponding to the soluble fatty acids. This volume multiplied by the factor 0.0088 gives the butyric acid in the weight of butter fat employed.[1]

The flask containing the cake of insoluble fatty acids is thoroughly drained and then placed on the water-bath to melt the contents, which are poured as completely as possible into the (wet) filter, through which the aqueous liquid was previously passed. The fatty acids are then washed on the filter with boiling water, to remove the last traces of sparingly soluble acids. The filter is then placed in a small dry beaker and treated in the manner described on page 38, the main quantity of fatty acids and the supplementary portion subsequently dissolved out of the flask and filter being weighed separately.[2]

When it is only required to determine the insoluble acids of butter fat the foregoing tedious mode of operating may be avoided by diluting the soap solution obtained by saponifying 5 grams of the fat till it measures about 300cc. The large excess of alkali is then neutralized by cautious addition of hydrochloric acid, and the hot solution treated with a slight excess of barium chloride or magnesium sulphate. The precipitated barium or magnesium soap is well washed with hot water, and then rinsed off the filter into a separator, where it is decomposed by dilute hydrochloric acid. The resultant layer of insoluble fatty acids is washed by agitation several times with warm water, and is then treated as directed on page 38.

In the analysis of butter fat, the sum of the insoluble fatty acids by weight and of the soluble fatty acids, calculated as butyric acid, should always amount to fully 94 per cent. of the fat taken. In the author's own experience the sum more frequently approaches or even exceeds 95 per cent., especially if the butter be adulterated.

The soluble fatty acids, calculated as butyric acid, should amount to at least 5 per cent., any notably smaller proportion being due to adulteration.[3] The insoluble fatty

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[1] Thus, suppose an experiment to have given the following figures: Weight of butter fat taken, 5.120 grams; decinormal soda required in the blank experiment, 3.90cc.; decinormal soda required to neutralize one-fifth of the solution of the soluble fatty acids, 6.25cc.; then

$$\frac{.008\ (31.25-3.9)\times 100}{5.120}=4.70 \text{ per cent.}$$

[2] Instead of weighing the insoluble fatty acids, W. F. Perkins has proposed to dissolve them in alcohol, and titrate with standard alkali in the manner described on page 76. The objection to this plan is the somewhat variable character of the fatty acids themselves. Calculating their neutralizing power on the assumption that they are wholly stearic acid, Perkins found 92.0 and 91.7 per cent. of insoluble acids in pure butter fat. Calculated to oleic acid these figures would not be materially modified, but their equivalents in palmitic acid are 83.3 and 83.0 per cent. respectively.

[3] According to J. Bell, the proportion of soluble acids calculated as butyric acid not unfrequently falls as low as 4.5, and the percentage of insoluble acids sometimes slightly exceeds 89.0.

acids from genuine butter fat rarely exceed 88½ per cent, occasionally reaching 89 per cent., but a sample ought scarcely to be regarded as certainly adulterated unless the insoluble acids exceed 89½ per cent. As a standard for calculation 88 per cent. of insoluble acids[1] may be regarded as a fair average, the soluble acids being taken at 5½ per cent.

Allen, in a later contribution to the literature of Reichert's method, says:[2]

A further experience in the employment of Reichert's process for examining fats has led me to abandon the expression of the results in terms of butyric acid, in favor of a statement of the weight of caustic potash neutralized by the distillation from 100 grams of the oil. This is obtainable by multiplying the volume of decinormal alkali neutralized by the distillate from 2.5 grams by the factor 0.2244.[3]

The following table contains a number of results expressed in both ways:

	C. C. of $\frac{N}{10}$ alkali required by 2.5 grams.	KOH required by 100 parts of oil.	Observer.
Butter or milk fat;			
Cow's	12.5 to 15.2	2.80 to 3.41	Reichert, Caldwell, Moore, Allen, &c.
Ewe's	13.7	3.07	Schmitt.
Goat's	13.6	3.05	Do.
Porpoise's	11.3	2.51	Allen.
Cocoanut oil	3.5 to 3.7	0.78 to 0.83	Reichert, Moore, Allen.
Palm-nut oil	2.4	0.54	Allen.
Palm oil	0.8	0.18	Moore.
Cacao butter	1.6	0.36	Do.
Butterine and oleomargarine	0.2 to 1.6	0.04 to 0.36	Caldwell, Moore, Allen.
Whale oil	3.7	0.83	Allen.
Do	12.5	2.80	Do.
Porpoise oil	11.0 to 12.0	2.47 to 2.69	Do.
Sperm oil	1.3	0.29	Do.
Bottle-nose oil	1.4	0.31	Do.
Menhaden oil	1.2	0.27	Do.
Cod-liver oil	1.1 to 2.1	0.24 to 0.47	Do.
Sesame oil	2.2	0.48	Do.
Cotton-seed oil	0.3	0.07	Moore.
Castor oil	1.4	0.31	Allen.

From these results it is evident that the fats of different kinds of milk (butter fats) are sharply distinguished from nearly all other fats by the large proportion of soluble volatile fatty acids they yield by Reichert's process. The most remarkable exception is presented by porpoise oil and some samples of whale oil. In porpoise oil I have found 5 per cent. of valeric acid, and Chevreul obtained as much as 9.63 per cent. In a recent paper I pointed out that in porpoise butter the glyceride of valeric acid appeared to replace the butyrin characteristic of the butter of terrestrial mammals.

Some of the chemists who have employed Reichert's process take the precaution to filter the distillate before titrating it, so as to get rid of any volatile acids which may be insoluble or very sparingly soluble in water. This plan may sometimes be adopted with great advantage. Thus when the solution of the soap obtained by saponifying cocoanut or palm-nut oil is acidulated and distilled, a notable proportion of lauric acid passes over and solidifies in the condenser or on the surface of the distillate; and

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[1] The percentage of adulterant in a butter fat may be calculated from the following formula, in which F. is the percentage of foreign fat and 1 that of the insoluble fatty acids: $F = (1-88) \times 13.3$. Or each 0.1 per cent. of soluble acids above 0.5 may be regarded as showing the presence of 2 per cent. of butter fat.

[2] Analyst, 1887, pp. 11 et seq.

[3] 1 cc. of $\frac{N}{10}$ alkali contains 0.00561 gram of KOH; and $\frac{.00561 \times 100}{2.5} = .2244$

by adding water to the contents of the retort, again distilling, and repeating this process several times, a very considerable proportion of volatile fatty acids can be obtained from cocoanut oil. In assaying butter, the appearance of insoluble acids in the distillate would furnish a valuable indication of the presence of cocoanut oil, and they should be removed by filtration, or the distillate will be found to neutralize so large a volume of alkali as considerably to diminish the practical value of the process as a means of distinguishing butter from butter substitutes, as has been pointed out by Moore and others. Latterly, I have adopted the plan of filtering the distillate in all cases, washing the filter with cold water, and then immersing the filter, with any adhering insoluble acids, in alcohol, which is then titrated with decinormal alkali and phenol-phthalein. In the case of ordinary butters and butter substitutes the insoluble volatile acids only neutralize about 0.2cc. of decinormal alkali.

The question having recently been raised, the following experiments were made at my request by Mr. William Barraclough on a sample of butter fat, in order to ascertain the variation in the results of Reichert's process produced by modifications in the methods of conducting the saponification and distillation:

(1) Two and a half cubic centimeters of butter fat was saponified by alcoholic potash in an open basin, the alcohol evaporated off completely at a steam heat, the residual soap dissolved in water, the solution acidulated with sulphuric acid in slight excess, diluted to 75cc. and distilled gently in a globular flask with side tubulure adapted to a condenser until 50cc. had passed over. The flask held 460cc. up to the side tube, and some fragments of pumice-stone coiled round with platinum wire were added to the contents to promote evolution of vapor.

(2) An exact repetition of No. 1 experiment.

(3) Saponification was effected in a flask furnished with a long tube and heated by steam. The subsequent manipulations were the same as in experiment 1.

(4) Saponification was effected in a well-closed bottle placed in the water oven. Other manipulations unchanged.

(5) Manipulation exactly as in experiment 3, except that the distillation was conducted in a flask fitted to the condenser by a cork and bent tube.

(6) Conducted as in experiment 3, except that the distillation was conducted in a retort.

(7) Blank experiment with the alcoholic potash employed in the previous experiments, the manipulation being that in experiment 3. The alcoholic potash was brown and not very recently prepared.

Experiments.

	Decinormal alkali for 2.5 grams.
	cc.
No. 1	11.80
No. 2	11.85
No. 3	12.40
No. 4	12.50
No. 5	12.40
No. 6	12.45
No. 7	0.25

These results show that a sensible loss occurs if the saponification be conducted in an open basin, doubtless owing to the formation of butyric ether. On the other hand, the exact nature of the distilling apparatus appears to be of little importance. This latter conclusion is not in accordance with the experience of some other chemists.

Zulkowsky and Groger[1] have studied and modified Haussman's method[2] of volumetric fat analysis. This method is based on the fact

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[1] Ber. Chem. Gesel., vol. 16, p. 1140.

[2] Ding. Poly., J., vol. 244, p. 303, and vol. 246, p. 286.

that an alcoholic solution of a fat acid is immediately saponified by the addition of alcoholic potash, while a neutral fat requires time and heat to secure complete saponification.

When, therefore, an alcoholic solution of fat acids and neutral fats to which phenol-phthalein has been added is titrated with caustic potash, the red color only appears when the fat acids are saponified, and only comes permanently when all the fats are saponified. When the red color appears an excess of caustic potash is added and the whole boiled for half an hour to saponify all the neutral fats and retitrated, whereby the amount of caustic potash required to effect the saponification of all the fats is ascertained, and the quantity of potash required for each titration represents the relative proportion of fat acids and neutral fats in the mixture operated on. When a neutral fat is saponified the following reaction takes place:

$$C_3H_5\,(C_nH_{2n-1}O_2)_3+3\ KOH=C_3H_5\,(OH)_3+3\ K\,(C_nH_{2n-1}O_2)$$

and therefore every litre of normal potash splits up one-third equivalent of glycerine, *i. e.*, 30.667 grams. One cubic centimeter normal potash is therefore equivalent to 0.030667 gram glycerine. The theoretical yield of fat acids could also be calculated by the following formula:

$$C_3H_5\,(C_nH_{2n-1}O_2)_3=\cancel{C_3H_5C_nH_{2n-1}O_2}.$$

Then one litre normal potash represents one-third equivalent of glycerine residue, or 12.667 grams. If 5cc. normal potash have been employed the weight of the glycerine residue would be .012667 × 5.

F. W. A. Woll[1] gives the results of his studies with butter and artificial butter.

Mixtures of pure butter with "oleo oil" were made and examined by the methods of Koettstorfer and Reichert, and the results compared with theory. The following numbers were obtained:

Per cent. butter.	Koettstorfer.			Reichert.		
	Calculated.	Found.	Difference.	Calculated.	Found.	Difference.
	mg.	*mg.*	*mg.*	*cc.*		*cc.*
20	200.8	201.4	+0.6	2.98	3.11	+0.13
40	206.2	207.3	+1.1	5.81	6.39	+0.58
50	208.5	209.0	+0.5	6.55	7.08	+0.53
60	211.5	212.7	+1.2	8.65	9.00	+0.41
80	217.7	215.6	−2.1	10.37	11.56	+1.19
Means	....	....	1.1	....	....	0.57

NOTE.—It is very easy to get exact results by the above method of mixture. Butter and an oil are used whose behavior with the reagents employed was determined by preliminary experiment. The case is very different when the analyst is called on to examine an *unknown* sample. The butter in an unknown sample may have quite a different per cent. of volatile acid from that used in the samples given. The value of this method, therefore, is seriously impaired for determining the extent of adulterations in case where the separate examination of the constituents is impossible.

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[1] Zeit. Anal. Chem., 1884, p. 28, and Am. Chem. Jour., vol. 9, p. 62.

The author gives a table of the analyses of 37 samples of butter and butter substitutes giving the percentage of water, the specific gravity at 37°.7 C., the melting point determined by the method of Blyth, the milligrams of KOH required in Koettstorfer's method and of cubic centimeters by Reichert's method.[1]

The author concludes that the melting point is of no value in discriminating between pure and false butters, but the specific gravity, the saponification process, and the distillation of the volatile acid are sufficient to distinguish at once between the true and the false.

The oleo oil employed had a mean specific gravity at 37°.7 C. of 0.90369 and its melting point was 27°.6 C. The "neutral" had a specific gravity of 0.9053 and a melting point of 38°.1 C.

BEHAVIOR OF COCOANUT OIL WITH SOME OF THE METHODS USED IN ANALYSIS OF BUTTER FATS.

R. W. Moore, in a paper read before the American Chemical Society, September 18, 1885,[2] calls attention to cocoanut oil as a substitute for butter. He gives its fusing point at 24°.2 C. to 24°.3 C., and calls attention to the fact that its specific gravity is higher than that of butter fat. It is also noticed that the insoluble acids in butter fats may sometimes amount to as much as 90 per cent.[3]

The author has found that cocoanut oil yielded 86.43 per cent. insoluble acids,[4] and thus infers that it could be mixed with other fats and escape detection by this method, calling attention to the fact, however, that if the soluble acids be estimated according to the method of Dupré[5] the sophistications might at once appear.

The low figures obtained are ascribed to the volatility of lauric acid which escapes on drying the insoluble fats. By Koettstorfer's process the number of milligrams potash necessary to saponify one gram cocoanut oil was found to be 257.3 to 258.3[6] the large quantity required being due to the presence of lauric, caproic, capryllic, and capric acids. It is, therefore, possible to mix oleomargarine and cocoanut oil in such a manner as to produce results similar to those given by pure butter. This is shown by the following figures:

Cocoanut oil.	Oleomargarine.		
Per cent.	*Per cent.*		*Milligrams.*
49.3+	50.7	required of KOH	220.0
70.2+	29.8	required of KOH	234.9
53.1+	46.9	required of KOH	223.6
75.9+	24.1	required of KOH	234.9

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[1] *Op. cit.*, pp. 31, 62, 63.

[2] Analyst, 1885, p. 224 *et seq.*

[3] Fleischmann and Veith, Zeit. Anal. Chem., 1878, p. 287; Kretschmar, Ber. Chem. Gesel., vol. 10, p. 2091; Kuleschoff, Wag. Jahresbericht, 1878, p. 999; Jehn, Archiv der Pharm., vol. 9, p. 335; De la Source, *Ibid.*, vol. 12, p. 929.

[4] Chem. News, vol. 50, p. 268.

[5] Analyst, 1877, pp. 87 and 114.

[6] Valenta, Ding, Poly. J., vol. 249, p. 270; Moore, Chem. News, *loc. cit.*

The oleomargarine used required 193.5 milligrams KOH per gram.[1] The iodine method of Hübl[2] was also tried with a mixture of cocoanut oil and oleomargarine, &c., and numbers obtained which come within the limits for pure butter.

Thus oleomargarine 55 per cent. + cocoanut oil 45 per cent. required 35.5 of iodine per 100 grams, and lard 40 per cent. + cocoanut oil 60 per cent. required 32.2 of iodine. In samples of butter the iodine numbers found by Hübl varied from 26.8 to 35.1.

By Reichert's method, however,[3] the presence of cocoanut oil mixed with butter and oleo is at once detected. Thus a mixture of 50 per cent. butter, 27.5 per cent. oleomargarine, and 33.5 cocoanut oil gave by Hehner's method 89.50 per cent. insoluble acids; by Koettstorfer's method, 227.5 mg. KOH; by Hübl's method, 35.4 per cent. iodine factor; by Reichert's method, 8.7cc. $\frac{N}{10}$ soda solution.

Pure butter requires by Reichert's method about 13cc., $\frac{N}{10}$ alkali to neutralize the volatile acids distilled over, while cocoanut oil in similar circumstances requires only 3.7cc. Little evidence is forthcoming in respect of the use of cocoanut oil as an adulterant of butter. It has been mentioned as an adulterant of lard[4] and Dietszch[5] mentions it as a compound of "Schmalzbutter." In attempts to use it as an adulterant of butter no great success was secured, since the oil not having been properly purified made the butter unpalatable. The smell and taste of the oil can be removed by a patent process of Jeserich and Meinert[6] which consists in treating the oil with superheated steam and saponifying any free fatty acids by calcined magnesia. The author closes his paper by recommending Reichert's process as superior to all others in examining for the purity of butters.

USE OF COTTON-SEED OIL AS A BUTTER ADULTERANT.

Cotton-seed oil is used largely as an adulterant for lard and butter. It has saponification equivalent of 285 to 296 and specific gravity at 99° C. .872, pure butter fat at the same temperature being .868.

Its further properties are thus described by Allen:[7]

> The oil as expressed from the seeds contains in solution, often to the extent of 1 per cent., a peculiar coloring matter, which is characteristic of this oil and its seed, and which gives the oil a ruby-red color, sometimes so intense as to cause the oil to appear nearly black. Crude cotton-seed oil gives a very bright red coloration with strong sulphuric acid (page 59). When boiled with an alkaline solution, alcoholic potash being preferable for laboratory experiments, crude cotton-seed oil is saponified, and the resultant soap rapidly oxidizes on exposure to air, with production of a fine pur-

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[1] Moore, Am. Chem. J., vol. 6, p. 416.

[2] Ding, Poly. J., vol. 253, p. 281.

[3] Zeit. Anal. Chem. 1880, p. 68.

[4] Analyst, 1882, p. 193.

[5] Nahrüngsmittel und Getränke, p. 212.

[6] Wag. Jahresbericht, 1882, p. 932.

[7] *Op. cit.*, 2d ed., p. 112.

ple or violet blue coloration.[1] This reaction is characteristic of crude cotton-seed oil. The coloring matter causes crude cotton-seed oil to produce stains, and hence is removed by a process of refining. This is usually effected by agitating the crude oil at the ordinary temperature with 10 to 15 per cent. of solution of caustic soda of 1.060 specific gravity, when the alkali combines with the coloring matter and saponifies a portion of the oil. The mixture becomes filled with black flocks which deposit on standing[2] and leave the oil but slightly colored. The loss in refining is usually from 4 to 7½ per cent., but occasionally amounts to 12 or 15. Hence it is desirable, before purchasing crude cotton-seed oil for refining, to ascertain, by a laboratory experiment, what the percentage of loss is likely to be. Frequently the treatment with alkali is only carried far enough to remove the major part of the coloring matter, the oil being then boiled with a solution of bleaching powder, and subsequently treated with dilute sulphuric acid.[3]

Refined cotton-seed oil is of a straw or golden-yellow color, or, occasionaly, nearly colorless. The density ranges from .922 to .926, and the solidifying point from 1° to 10° C. By subjection to cold and pressure a certain proportion of stearine is separated, the melting point of the residual oil being correspondingly lowered. Refined cotton-seed oil is usually very free from acid, and when properly prepared is of pleasant taste and admirably adapted for edible and culinary purposes, for which it is now extensively employed, both with and without its nature being acknowledged. It is now substituted for olive oil in some of the liniments of the United States Pharmacopœia, but its principal applications are in soap making and the manufacture of factitious butter.

ESTIMATION OF SALT.

The method employed in this laboratory since 1883 has continued to give satisfaction, and can be recommended as the best in use.

From 10 to 20 grams of the well-mixed butter or butter substitute are placed in a separatory bulb provided with a closely fitting glass stopper. Add 25 to 50cc. hot distilled water, and after shaking well allow to stand for a few minutes. The water, which has dissolved most of the salt, is now drawn off through the stoppered tube of the apparatus. Fresh hot water is added and this operation repeated until the

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[1] "Cotton-seed blue" is stated by Kuhlmann to have the composition of $C_{17}H_{24}O_4$. It is amorphous, readily destroyed by oxidizing agents, insoluble in water, diluted acids, and alkalies, sparingly soluble in carbon disulphide and chloroform, but more readily in alcohol and ether, and dissolves with purple color in strong sulphuric acid. The unoxidized coloring matter of cotton-seed oil has been recently examined by J. Longmore, who, in a communication to the author, states that it is a pungent golden-yellow product, insoluble in water, but soluble in alcohol and alkaline solutions, and precipitated from the latter on addition of acids. It dyes well and perfectly fast on both wool and silk.

[2] The deposit thus formed, consisting of coloring and albuminous matters, alkali, and partially saponified oil, is technically called "mucilage." It is decomposed with a slight excess of acid, and the resulting dark-colored grease is heated to a temperature of 120° C. (=250°F.) with concentrated sulphuric acid, which renders insoluble the coloring matters, &c., while the impure fatty acids rise to the surface. On distilling these with superheated steam, a mixture of fatty acids is obtained, which is separated into stearic and oleic acids by pressure. The "cotton-seed stearine" thus obtained is employed for making soap and composite candles, as also for adulterating tallow, &c.

[3] This method of treatment is economical, but causes the oil to acquire an unpleasant taste and smell, which cannot be removed.

volume of the wash water amounts to 250 to 500cc. By this time all the salt has been dissolved and separated from the butter.

Chromate of potassium is now added to the salt solution, and the titration is accomplished by a standard silver nitrate solution.

The amount of NaCl in butter is also determined by dissolving the fat with ether or light petroleum, and after incineration of the curd, weighing the residual ash, which is taken as the amount of salt present. This method is not to be recommended since it includes the salt found in the other mineral constituents.

Sell[1] gives the following method: Ten grams of butter are weighed into a porcelain crucible and dried at 100° C. for six hours. The melted fat, &c., is now filtered, and crucible and filter are washed with ether. The filter with its contents is then incinerated. The ash is extracted with water, filtered, and the NaCl estimated volumetrically in the filtrate.

ESTIMATION OF CURD.

The methods of estimating curd depend on the principle of first drying a weighed portion of the butter, and afterwards extracting the fat with ether or petroleum. The residual mass is then weighed and the curd determined by loss on ignition. This process is carried on in this laboratory as follows:

Five to ten grams of butter are dried at 100° C. for a few hours in a porcelain dish. The dried fat, &c., are filtered through a Gooch crucicle, the contents of the dish all brought into the crucible and well washed with ether or light petroleum. The filter crucible is dried for two hours and weighed. The curd is then determined by loss of weight on ignition. A number of experiments have also been made to convert the curd directly into an ammonium compound by Kjeldahl's process. This method has not met with sufficient success to merit a recommendation to general use. This method was first tried in the laboratory in 1884.

Babcock finds this method more satisfactory.[2] Ten grams of the fat are treated with light petroleum, and after the fat solution has been decanted the treatment is repeated. The purified curd is then treated by Kjeldahl's process.

QUALITATIVE TESTS.

The qualitative tests employed in the detection of artificial butter are the following:

(1) Microscopic examination. This method has already been sufficiently described.

(2) Solubility in a mixture of amyl-alcohol and ether.

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[1] *Op cit.*, p. 527.

[2] Fifth Ann. Rept. Bd. Control N. Y. Exp. Sta., p. 335.

The quantity of stearin in butter fat is small compared with that in lard, tallow, &c. On this difference of constitution Professor Scheffer[1] has based a method of analysis.

A mixture is made containing 40 volumes of rectified amyl-alcohol and 60 volumes ether of .725 specific gravity at 15° C. One gram of butter fat is dissolved in 3cc. of this mixture at 26° to 28° C. On the other hand, 1 gram lard requires 16cc. of the solvent, 1 gram tallow 50cc., and 1 gram stearin 350cc.

For the experiment take a test tube of 12cc. capacity and place in it 1 gram fat, add 3cc. of the amyl-alcohol ether mixture. After tightly corking the tube put it in a water bath of 18° C. and with frequent shaking bringing the temperature to 28° C. If the butter is pure the solution becomes perfectly clear at this temperature. If not clear more of the solution can be run in out of a burette and the additional quantity required will be some indication of the quantity or quality of the adulterant which has been used.

According to Scheffer, mixtures of pure butter and lard gave the following data:

Butter.	Lard.	Quantity of mixture required.
Gram.	*Gram.*	*CC.*
.1	....	3.0
.9	.1	3.9
.8	.2	4.8
.7	.3	5.7
.6	.4	6.5
.1	.9	14.4

A trial of this method has shown that it is capable of giving valuable qualitative indications in respect of the purity of the sample under examination. I believe it is the best simple test aside from the microscopic examination capable of general application which has been proposed.

The easiest method to secure a certain weight of fats is to melt them and measure out from a pipette 1 cubic centimeter of each. The fats which do not melt easily should be stirred up thoroughly with a wire, while the temperature is raised from 18° to 28° C.

(3) Odor of the burning grease.[2]

(4) The insolubility of the stearate of potash in alkaline solutions.[3]

(5) Insolubility of tallow, lard, &c., in petroleum ether of .69 specific gravity.[4]

(6) The relative solubility of butter fats and substitutes therefor in a mixture of 50 per cent. alcohol and 66 per cent. ether.[5]

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[1] Pharm. Rundsch., 1886, p. 248.

[2] Kunstmann. Pharm. Centralh., 1875, No. 9.

[3] Gatehouse, Chem. News, vol. 32, p. 297.

[4] Zeit. Anal. Chem., 1872, p. 334.

[5] Husson Zeit. Anal. Chem., 1880, p. 236; Filsinger, Pharm. Centralh., 1878, p. 260.

(7) Crook[1] warms half a gram of the filtered fat in a test tube to 66° C., and adds 1.5cc. phenol, shakes and warms in water bath until the liquid is clear. On standing pure butter gives a homogeneous solution· Tallow and lard appear, however, in distinct layers.

A method somewhat similar to this was proposed in 1877 by Bach.[2] The apparatus required consists of a test tube and a thermometer. The reagent is a mixture of 3 volumes ether and 1 volume alcohol of 95 per cent. and 1 gram of the butter or tallow and put in the test tube with 20cc. of the above mixture, and this is placed in water at 20° C. At this temperature pure butter is completely dissolved. Butter, however, containing lard, beef, or mutton tallow remains undissolved.

(8) Horsely[3] calls attention to the perfect solubility of pure butter in ether, and that it is not precipitated from this solution by methyl-alcohol, while other common fats are thus separated at 20° C.

Leuz[4] confirms the general results of the foregoing process.

(9) Belfield[5] allows the fats dissolved in ether to crystallize, and distinguishes between them by their crystalline form.

(10) Paillat[6] has found that pure butter when mixed with copper oxide in ammonia gives a turquois blue color, while a butter adulterated with margarine (?) gives a greenish tint.

(11) Dubois and Padé[7] point out that the addition of any considerable quantity of foreign fats to butter not only changes the melting point of the fatty acids obtained, but also diminishes their solubility in alcohol.

(12) Wolkenhaar[8] distinguishes between the different fats by means of nitric acid, which gives to cotton seed oil, palm oil, lard, sesame oil, and several others a red brown color.

For a fuller discussion of most of these qualitative tests, consult either the original articles or Sell.[9]

(13) Method of Mayer.[10] This test is made as follows:

About 0.6 gram of butter fat is placed in a test tube with 12cc. water made slightly alkaline by a few drops of a solution of 2 per cent. soda, or two drops of 6 per cent. ammonia-water. The tube closed by the thumb is then well shaken, afterwards carried to a temperature of 37° C. to 40° C., with frequent shaking. The emulsion thus formed is poured into a separatory funnel. The fat is now washed several times with water at 37° C. to 40° C., the wash-water being drawn off by the stop-cock so as to maintain a constant level in the funnel. The fatty matter having thus been placed in contact with about 400cc. water, the stop-cock is so

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[1] Analyst, 1879, p. 111.

[2] Pharm. Centralb., 1877, p. 166.

[3] Chem. News, vol. 30, p. 135 and 154.

[4] Zeit. Anal. Chem., 1880, p. 370.

[5] Rep. d. Ver. Anal. Chem., vol. 3, p. 383.

[6] L'Année Scientifique par Louis Figuier, 29th year, 1885.

[7] Bul. Soc. Chim., vol. 44, p. 602.

[8] Rep. d. Ver. Anal. Chem., vol. 3, p. 103.

[9] *Op. cit.*, pp. 505–509.

[10] Jour. de Pharm. et de Chim., vol. 15, p. 97.

adjusted as to allow the removal of the wash-water as completely as possible. After cooling, the fatty matter remaining on the sides of the funnel is examined. If the butter be pure, there will be seen only a finely-divided mass, but the addition of a small portion of other fats will be revealed by greasy drops, which can be seen even during the progress of the washing. Natural butters made in summer require a lower temperature for the washing, viz, 35° C. to 37° C.

In most cases the microscopic test with polarized light and selenite plate combined with the solubility of the fat in the ether amyl-alcohol solutions will be found sufficient for the qualitative examination of a suspected butter.

RESULTS OF ANALYSES OF GENUINE AND SUSPECTED BUTTERS AND BUTTER ADULTERANTS.

TABLE NO. 6.—*Analyses of butter.*

Serial number.	Specific gravity at 40° C.	Water.	Insoluble acid.	Soluble acid, by washing out.	Soluble acid, by distillation.	Salt, NaCl.	Albuminoids.	Curd.	Koettstorfer's equivalent.	Vol. $\frac{N}{10}$ soda for 2.5 grams.
		Pr. ct.	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*		
1742	.91046	13.33	88.64	4.01	4.50	2.84	.7875	1.46	254.20	12.50
1743	.91119	8.53	87.85	4.14	4.57	3.69	.8312	1.31	250.60	13.10
1744	.91032	8.57	88.65	3.52	4.78	2.81	.8750	1.30	268.50	13.50
1745	.91067	8.14	88.08	3.68	5.48	2.04	.5688	1.25	264.90	15.30
1746	.91029	16.82	88.91	3.00	4.56	3.79	.7438	1.50	252.70	12.90
1747	.91244	4.59	86.60	5.02	5.51	3.41	.5250	.83	244.30	15.60
1749	.91165	11.46	87.50	5.49	4.61	1.48	.8312	1.14	250.10	13.10
1752	.91004	17.38	88.07	3.70	4.54	0.00	.4375	0.68	238.60	12.80
1759	.91013	13.95	87.47	4.73	4.80	0.06	.4375	0.81	249.70	13.00
1760	.91063	22.12	87.84	4.98	4.70	0.00	.1750	0.49	248.70	13.40
1761	.91067	23.46	87.47	5.27	4.99	0.00	.1750	0.59	243.06	14.10
1762	.91089	21.02	87.38	5.15	4.93	0.00	.2188	1.01	248.86	14.10
1763	.91073	11.89	87.71	4.69	4.98	2.61	.2625	1.30	244.90	14.10
1764	.91155	21.96	86.65	5.34	4.74	0.00	.4375	1.21	244.00	13.20
1765	.90958	31.55	88.09	4.45	5.02	0.57	.6125	1.83	252.00	14.30
1766	.91042	11.17	....	5.31	4.52	2.56	.4375	1.11	247.00	12.80
1768	.90995	7.68	87.24	5.08	5.21	5.62	.2625	0.71	247.00	14.80
1769	.91183	9.68	87.30	5.94	5.05	4.09	.5230	1.37	244.10	14.30
1770	.91069	7.35	88.14	5.05	4.47	5.28	.4375	0.91	252.10	12.70
1771	.91079	12.28	87.00	5.37	4.93	3.69	.4813	1.08	240.40	14.00
1772	.91093	8.89	87.21	5.47	5.20	3.18	.3063	1.03	245.10	14.90
1773	.91064	18.75	86.68	4.75	4.03	0.00	.7000	1.41	260.70	11.40
1775	.91034	9.87	87.58	5.17	4.56	4.83	.4375	1.12	251.80	12.90
1776	.91239	10.84	86.61	5.42	4.45	3.12	.4375	0.97	250.90	12.70
1777	.91031	12.28	88.48	4.06	3.92	5.79	.7438	1.43	236.50	11.10
1781	.91010	7.26	....	3.97	......	6.42	.4375	1.43	247.10	13.20
1782	.91112	12.32	87.23	4.24	......	6.53	.5250	2.02	245.40	13.60
1783	.91082	6.03	87.59	3.92	......	3.92	.5250	1.33	248.40	12.50
1785	.91186	8.29	87.10	4.48	......	5.11	.4375	1.16	247.50	14.50
1789	.91061	8.44	87.73	3.91	......	3.15	.7000	1.42	246.60	12.60
1790	.91080	4.44	87.85	4.41	......	1.81	.7000	1.02	251.50	13.90
1792	.91106	13.07	88.25	3.47	......	7.10	.4375	3.10	240.20	12.30
1795	.91136	8.22	87.75	4.18	......	4.37	.5125	1.34	210.70	14.50

TABLE NO. 7.—*Analyses of doubtful butters.*[1]

Serial number.	Specific gravity at 40° C.	Water.	Insoluble acid.	Soluble acid, by washing out.	Soluble acid, by distillation.	Salt, NaCl.	Albuminoids.	Curd.	Koettstorfer's equivalent.	Vol. $\frac{N}{10}$ soda for 2.5 grams.
1748	.90968	7.45	89.45	3.61	4.60	2.64	.7443	1.41	252.80	13.10
1757	.90064	11.30	89.44	3.54	4.25	5.28	.5688	1.63	253.60	12.10
1758	.90987	12.12	87.60	4.71	4.54	0.00	.4375	0.93	251.50	12.90
1767	.90974	10.90	88.68	4.73	4.45	2.16	.4813	1.33	249.70	12.60
1774	.90972	29.84	87.82	4.84	4.27	0.00	.9625	1.86	260.10	12.10
1779	.90947	11.59	88.01	3.16	......	5.00	.8750	1.50	250.60	12.50
1780	.90064	10.06	88.42	3.02	......	5.40	.8750	1.58	250.70	11.60
1793	.90938	8.50	88.00	3.31	......	13.00	....	1.12	253.50	12.30
1794	.90965	9.06	88.50	3.44	......	2.84	.4375	0.98	252.00	11.70

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[1] These samples were bought for pure butter, but, on analysis, ~~proved~~ to contain adulterants.

TABLE NO. 8.—*Analyses of butter substitutes.*

Serial number.		Specific gravity at 40° C.	Water.	Insoluble acid.	Soluble acid, by washing out.	Soluble acid, by distillation.	Salt, NaCl.	Albuminoids.	Curd.	Koottstorfer's equivalent.	Vol. $\frac{N}{10}$ soda for 2.5 grams.
			Pr. ct.	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*		
1750	Lard	.90538	0.00	92.59	0.41	0.08	0.00	.0875	Trace.	294.30	0.20
1751	Beef suet	.90158	0.00	92.59	0.22	0.04	0.00	....	0.01	296.90	0.10
1753	Oleomargarine	.90490	9.34	93.59	0.12	0.25	3.04	.3500	0.03	274.00	0.70
1754	Neutral lard	.90309	7.42	90.00	0.20	0.10	0.49	....	0.02	270.50	0.30
1755	Creamery butterine[1]	.90569	11.69	92.90	1.16	1.53	2.39	.3063	0.74	274.80	4.30
1756	Oleo fat[2]	.90287	14.23	93.35	0.10	0.08	0.97	....	0.60	280.10	0.20
1787	Country print	.90561	14.45	93.72	0.09	......	2.42	.8750	1.82	281.10	1.90

[1] 40 butter fat, 15 oleo fat, 30 neutral lard.

[2] Average 40 pounds per fat steer.

ANALYTICAL RESULTS.

The butters in table No. 6 were bought in open market and accepted as genuine on the results of the analysis. Some of these, however, ought justly to be classed in Table No. 7, as of doubtful purity. In quite a number of cases the number of cubic centimeters of decinormal alkali required to neutralize the distillate from 2.5 grams of the fat was less than 13. Nos. 1742, 1746, 1752, 1766, 1770, 1773, 1775, 1776, 1777, 1783, 1789, and 1792 come under this category. In all these cases, however, except 1765 and 1768, the specific gravity is above .910 at 40° C., and it would not be safe to condemn a butter as adulterated which had that specific gravity, unless the microscope should reveal crystals of foreign fat. In these samples such was not the case.

In the two cases mentioned, where the specific gravity fell below .910, there are other reasons for thinking the samples pure. In 1765 the percentage of soluble acid, by Reichert's method, is high, viz, 5.02. In 1768 it is still higher, viz, 5.21. With such proportions of soluble acid it would not be possible to condemn the samples as adulterated on the evidence of the specific gravity alone.

On the other hand, when the percentage of soluble acid is low, as in 1777, the specific gravity and saponification equivalent prevent the classification of the sample among the doubtful butters. Nevertheless, should such a sample show with polarized light and a selenite plate bi-refractive crystals, it would be a strong presumptive evidence of adulteration. In any case, such a sample as 1777 would present numerous difficulties to the analyst, especially if he were called to testify in respect to its purity.

In Table No. 7 similar difficulties are encountered. The specific gravities are uniformly low. On the other hand, the percentage of insoluble acids are only suspiciously high in two instances, viz, 1748 and 1757. In the first of these instances, however, the soluble acid is above the limit of suspicion. The saturation equivalent is uniformly rather high, but not above the range of pure butters. While the butters are classed for convenience as "doubtful," they could not be so proved before a court on the chemical evidence alone.

In Table No. 8 we have plain sailing. All analytical data show the fats of the samples examined are not butter. Since the adulteration of butters with less than 30 per cent. of a cheaper fat could scarcely prove profitable, the chemist should be careful not to condemn a suspicious sample, if its purity be attested by any one of the processes employed in the examination, unless some one test shows it to be undoubtedly adulterated.

In the foregoing study of methods of analysis I have not attempted to give a complete citation of all the papers which have been written on this subject. A very complete bibliography of the subject up to 1882 is given by Caldwell,[1] and in the work of Sell.[2]

The probability of the detection of an adulterated butter by the physical and chemical processes described in the foregoing pages is very great.

In the order of value the quantitative processes employed may be arranged as follows: (1) Determination of volatile acids by distillation. (2) Determination of specific gravity. (3) Determination of the saponification equivalent. (4) Determination of the insoluble acids. (5) Determination of the melting point.

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[1] Second Ann. Rept. N. Y. S. Bd. of Health, pp. 544–7.

[2] Arbeit a. d. Kaiserlichen Gesundheitsamte.

EXAMINATION OF MILK.

The adulteration of milk in this country consists usually either in the removal of cream or the addition of water.

Without making any attempt whatever to notice the prolific literature of this subject, which has accumulated during the past few years, such portions thereof as seem to be most helpful in the work of analysis will be cited. Those who care to study the subject in greater detail are referred to the periodical literature, especially to the "Analyst" and "Milch Zeitung."

The constituents of milk which are to be determined by analysis are (1) water; (2) sugar; (3) nitrogenous constituents; (4) ash, and (5) fat.

Water.—The simplest method for estimating water in milk consists in evaporating one or two grams in a flat platinum dish. The larger the diameter of the dish the quicker and more accurate will be the results.

If larger quantities of milk be used or the dish have not a flat bottom, the film which forms over the surface of the milk during evaporation will prevent complete desiccation. To avoid this many plans have been proposed. The milk may be mixed with gypsum, and then a larger surface be exposed and more rapid and complete drying secured.

Instead of gypsum, sulphate of barium, pure quartz sand, sulphate of strontium, and powdered glass have been used. All of these methods are capable of giving fairly accurate results when properly conducted.

The addition of acetic acid or alcohol to coagulate the albuminous matter before desiccation has been largely practiced, but Gerber and Radenhausen have shown[1] this treatment is without influence on the results. Jenks has also shown[2] that simple evaporation without any treatment whatever gives results which agree well with those obtained by using sand.

In fifty determinations the maximum and minimum difference between the two methods was only .14 per cent. and the mean difference .003 per cent.

Babcock[3] has proposed an ingenious and accurate method of determining the water in milk:

About two grams of rather coarse asbestos are placed in a platinum evaporator of 30cc. capacity, ignited and weighed. Five cubic centimeters of milk from the pipette,

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[1] Bied. Centralblatt, 1876, p. 22.

[2] Chem. Centralblatt, 1882, p. 13.

[3] Second Ann. Rept. Bd. Control N. Y. Exp. Sta., pp. 167–8.

previously weighed, is run into the evaporator and the pipette weighed again. The milk in the evaporator is then dried at 100° C., until the weights taken one-half hour apart do not vary more than a milligram from each other.

The asbestos serves as an absorbent of the milk and presents a large surface which greatly facilitates the drying. For this purpose asbestos is much to be preferred to sand or any fine powder which requires frequent stirring for complete desiccation. When a number of analyses are to be made in succession, a second portion of milk may be dried in the same asbestos with advantage. In the series of analyses made during the feeding experiments the morning's and evening's milk were dried together in this way. The dried residue may be ignited for ash.

The figures given for solids in all analyses made during the year have been determined in the above manner. The solids may, however, be found with equal accuracy and in much less time by the method given below.

In the bottom of a perforated test-tube, such as is used in the estimation of the fat in fodders, is placed a tuft of clean cotton. The tube is then filled three-quarters full of ignited asbestos and a plug of cotton inserted to prevent the escape of loose fibers of asbestos. The asbestos must be slightly pressed together so as to leave no large spaces. The tube and contents are weighed, the plug of cotton carefully removed, and five grams of milk, from the weighed pipette, described before, run into it and the plug of cotton replaced. The tube, connected at its lower end by a rubber tube and adapter with a filter pump, is placed in a drying oven at 100° C. and a slow current of dry air drawn through it till the water is completely expelled, which in no case requires more than two hours.

Since the publication of the method of Adams for the estimation of fat, which will be given further on, I have made some attempts to estimate the water by drying the milk on long strips of asbestos paper, which are rolled up while still hot and weighed after cooling in a dessiccator. I have not yet secured an asbestos paper sufficiently bibulous to make this method completely successful. But it has the advantage of being very speedy, since on so large a surface exposed for two or three minutes to a temperature of 100° to 105° C. over a sand bath the water is completely evaporated.

An indirect method of estimating the water from the specific gravity has been prepared by Behrend and Morgen[1] by the formula—

$$S^2 = \frac{S(V - A)}{V - S^1}$$

in which S = specific gravity of the milk, S^2 = specific gravity of the milk free of fat, S^1 = specific gravity of the milk fat = .94, and V = volume taken = 100cc.

Numerous tables are given by the authors to show the agreement between the calculated percentage of fat and total solids obtained by the above formula and the gravimetric determinations.

Another indirect method of determining the quantity of water in milk consists in measuring the quantity of finely-pulverized common salt a given volume of it will dissolve.

This procedure was proposed by Reichelt.[2]

The apparatus consists of a glass vessel 24cm. high. The upper part has a diameter of 2.5cm. and the lower of 8mm. On the under side is a

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[1] Jour. Landw., 1879, p. 249.

[2] Bayerish Kunst und Gewerbeblatt, 1860, p. 706.

scale marked to 45° C. The principle of the apparatus is based on the fact that at 30° to 35° C., 100 parts of water will dissolve 36 parts of salt. The operation is carried on as follows: Mix 62.5 grams of milk with 20.25 grams of salt and add 15 grams of litmus tincture, saturated with salt, to color the milk. Raise the temperature to 30° to 35° C., shake thoroughly, and then place the apparatus so that all the undissolved salt will fall into the under-graduated stem of the apparatus. Each degree of the scale corresponds to 62.5mgr. of the salt. The part undissolved subtracted from the total quantity will give the quantity dissolved, from which the quantity of water is easily calculated.

The lactometer of Geissler[1] is too complicated for ordinary use, and the method of estimating the water content of milk by measuring the volume of whey filtered from the coagulated albumens proposed by Zenneck[2] does not afford sufficiently exact results to merit further description.

SPECIFIC GRAVITY.

The specific gravity of a milk diminishes as its content of fat increases, and hence within certain limits it may be a valuable index of the character of the sample under examination.

When the cream has been removed, however, the specific gravity may be reduced to that of normal milk by the addition of water, and then the determination of the specific gravity alone is not a certain method of detecting adulteration, yet it is a valuable indication and should always be determined.

This determination may be made by any of the methods already denoted for fats and oils or by a hydrometer. Since the use of this latter instrument (lactometer, lactodensimeter) is easy and speedy, it is generally employed instead of the slower but more exact procedure with a picnometer.

Martin[3] found the average specific gravity of the milk from fifty cows from E. B. Brady's farm, Westchester, N. Y., to be 1.03101. From another lot of thirty one cows, farm of Peter Knox, it was 1.03149; from sixteen cows, farm of George Nelson, 1.03175.

Jenkins[4] makes the following observations respecting the values of the specific gravity determination:

> A consideration of the observations noticed above brings us to the following conclusions with regard to the value of *total solids*, and of *specific gravity*, as criteria for judging of the quality of milk.
>
> We have seen that pure herd-milk shows very wide variations in its content of solids and fat, and varaitions less striking in its specific gravity. No instance appears to be on record where a competent observer has found for the mixed milk of a number of healthy cows a specific gravity less than 1.029, and we may conclude with certainty that milk which falls below that density has been watered.

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[1] Ber. Chem. Gesel., Vol. 10, p. 1272.

[2] Vieth, Milchprüfungsmethoden, p. 87.

[3] Fourth Ann. Rept., N. Y. State Bd. of Health, pp. 429 *et seq.*

[4] Rept. Conn. Exp. Sta., Vol. 10, pp. 88, 89.

As evidence of watering simply, specific gravity furnishes by far the most satisfactory test, and if 1.029 is adopted as a minimum, no pure milk will be condemned. In some cases moderately watered milk may escape detection.

If we will establish a minimum limit for the percentage of solids and fat which shall in no case condemn pure milk in any locality, we shall have to make it absurdly low, and thus offer a premium on watering milk of good quality.

QUANTITY OF WATER OR DRY SOLIDS IN MILK.

The law of Massachusetts fixes the legal maximum of water in milk at 87 per cent. The quantity, however, varies within large limits, and it is manifestly unjust to condemn a milk as adulterated when it has more than 87 per cent. water.

The chief factors which cause a healthy milk to vary in its percentage of solids are length of time the cow has been in milk, the season of the year, and consequently the character of the food of the animal. On this point the Report of the Massachusetts State Board of Health[1] makes the following observations:

> The statutes prescribe a fixed and definite standard for commercial milk. Milk not containing 13 per cent. of solids is deemed to be adulterated under the law. It is often urged that, under such a standard, milk as obtained direct from the animal does not always conform to the requirements of the law. While this is true, it is also evident that a standard established at the minimum of quality, or that of the poorest milk obtained under the worst conditions, would admit of the sale of a very large quantity of adulterated milk.
>
> It is possible to produce from inferior animals, under unfavorable conditions, such as impoverished diet, bad care, extreme age or youth, milk somewhat below the legal requirement. This ought not to be an argument for the reduction of the standard to include occasional cases of the lowest quality.
>
> Mixed milk contains a greater amount of solids than its minimum constituents. Hence, the milk producer or dealer will find it a safe rule to sell mixed milk only, especially when his herd contains one or more animals producing milk of a poor quality. In the 40-quart cans of the Housatonic Valley, filled for the New York market, the milk must necessarily be a mixture from several animals, but in the case of the usual 2-gallon can, so largely in use throughout the larger part of this State, the contents may be often that of two or three animals only, and it occasionally may represent a single animal.

Under the Massachusetts law a rigid inspection of the milk sold in all the large cities is made, and the character of the milk is described in the following summary of the report of Dr. Harrington, milk inspector for Boston.[2]

> During the year just ended I have received from the inspectors of the board, and from other sources, 1,759 samples of milk, which number includes samples from all of the cities and many of the towns of Eastern Massachusetts. They have been arranged in classes, according to their respective sources, to wit: (*a*) Samples from shops; (*b*) samples from wagons; (*c*) samples from producers (direct); (*d*) samples from unknown sources; (*e*) samples of known purity.
>
> Among such a number, taken in most cases at random, there must necessarily be very many which, on inspection alone, are evidently pure, and which, on analysis, would yield figures above the standard fixed by law. The employment of the lacto-densimeter, together with the Feser lactoscope, will, after a little practice, enable

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[1] 1884, p. 116.

[2] Rept. Mass. S. Bd. of Health, 1884, pp. 145, 14

one to separate the good from the indifferent and poor samples, and in this way the good samples may, unless there be some reason for a full analysis—such, for instance, as unusual richness, averages, &c.—be passed on inspection. More than half of the samples submitted to me were good samples, and of the whole number 569 were passed as above, leaving 1,190 which were subjected to analysis. Of these latter there were 391 which were above, and 799 which were below, the statute standard.

Considering as above the standard the 569 samples which were passed on inspection, we have 960 above and 799 below, which is a very decided improvement over the milk supply of a year ago. A large proportion of those below the standard were not what would be considered as of very inferior quality; nearly one-half of those below the standard of 13 per cent. of solids were above 12 per cent., which fact of itself is evidence of a great improvement in the general supply.

Dr. E. W. Martin [1] has made a thorough study of milk adulteration, of which I give the following abstract:

Having made from time to time analyses of milk from cows of all breeds and kept under all conditions, of various ages, and at different times of the year, I found the percentage of the maximum, minimum, and average constituents to be:

Constituent.	Maximum.	Minimum.	Average.
	Per cent.	*Per cent.*	*Per cent.*
Water	82.04	87.87	87.5
Fat	7.59	2.78	3.3
Sugar	5.39	4.60	4.4
Caseine	4.34	4.30	4.1
Salts	.71	.65	.7
Total solids	17.96	12.13	12.5
Solids not fat	10.37	9.35	9.2

Of the minimum amount of fat given above, only three cows were found giving milk so poor in fat, and their food and surroundings were of the poorest kind.

During the year 1883 I made many analyses of milk taken from the cans while being shipped to market, and the average percentage of the constituents of the samples taken were:

	Per cent.
Water	87.5
Fat	3.2
Sugar	4.4
Caseine	4.1
Salts	.7
Total solids	12.5
Solids not fat	9.3

From the foregoing results it is fair to assume that in average milk we should have at least 3 per cent. of fat, 9.2 per cent. of solids not fat, and 12.2 per cent. of total solids.

The State Board of Health of New Jersey have fixed the minimum amount of total solids at 12 per cent. and the maximum amount of water at 88 per cent.

In Massachusetts the law fixes a chemical standard of purity; it reads: "In al cases of prosecution, if the milk shall be shown upon analysis to contain more than 87 per cent. of water or to contain less than 13 per cent. of milk solids it shall be deemed for the purpose of this act to be adulterated."

This standard has been fixed from analyses by Sharples, Babcock, and others, as follows:

Analysts.	No. of cows.	Total solids.
Sharples	22	14.49
Babcock	8	14.55
Vaughn	58	14.08
Newton	24	14.26

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[1] Fourth Ann. Rept. N. Y. S. Bd. of Health, pp. 429 *et seq.*

ESTIMATION OF FAT.

Gravimetric.—The percentage of fat in milk is the best criterion of its purity, although it is not impossible to make an emulsion with an added fat or oil after the natural fat of a milk has been removed.

For determining the quantity of fat in a milk by weight it is necessary to evaporate the sample to dryness and dissolve the fat by ether or a light petroleum.

Since, if the milk be evaporated in bulk, portions of the fat will be occluded by the other solids, it is necessary, in order to secure a total solution of the fat, to distribute it over some inert substances. Those already mentioned used for determining water, may also be employed for the fat analyses.

We use thin glass dishes (schälchen), in which the milk is dried in a thin film, or on sand, gypsum, or asbestos. The dish and its contents are then rubbed up in a mortar and transferred to a continuous extraction apparatus. The one employed is a modification of the continuous extractor made by Mr. A. E. Knorr, in which the return siphon is placed wholly within the extraction tubes, thus making the apparatus more compact and less liable to get broken.

The different methods of manipulation heretofore employed for the gravimetric determination are so well known that I will not describe them further, but pass at once to the consideration of a new process, which I have already tried sufficiently to show its merits over all others.

ADAMS'S METHOD.

Adams's method of estimating fat in milk[1] differs from the ordinary gravimetric methods solely in the preparation of the fat for extraction. Instead of drying the fat in the usual way the milk is absorbed by bibulous paper. It is unnecessary to state that this paper must first be thoroughly exhausted by the solvent which is used to dissolve the fat.

The kind of paper and the method of using it first proposed by Adams, are as follows:

> As for material, the only extra article is some stout white blotting-paper, known in the trade as "white demy blotting mill 428," weighing 38 pounds per ream. This should be in unfolded sheets, machine-cut into strips 2½ inches wide and 22 inches long; each sheet in this manner cuts into seven strips.
>
> I have tried other papers, but none have answered so well as this; it is very porous and just thick enough. Each of these strips is carefully rolled into a helical coil, for which purpose I use a little machine, made by myself, consisting of a stout double wire, cranked twice at right angles, and mounted in a simple frame. One end of the strip being thrust between the two wires, the handle is turned, and the coil made with great facility. This may be done, for the nonce, on a glass rod, the size of a cedar pencil. Two points have to be carefully attended to: the paper must not be broken, and the coil must be somewhat loose, the finished diameter being a little under an inch. I am in the habit of rolling up a considerable number at a time and

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[1] Analyst., 1885, pp. 46 *et seq.*

placing each within a brass ring as it is rolled, inscribing on one corner with a lead pencil its own proper number.

These coils are next thoroughly dried, and I need hardly say the accuracy of the process depends upon this drying. This can be satisfactorily done in an ordinary air bath at 100° C., providing the bath be heated properly and the paper kept in it long enough. I found the common way of heating the thin bottom of the bath with a single jet not to answer. My bath is placed upon a stout iron surface, which is heated by a large ring of jets; in this way the heat is evenly distributed over the whole of the bottom of the bath, and the papers, which are put in a cage frame of tinned iron wire 5 by 2½ inches and divided into eight partitions, get evenly and completely dried, if allowed to remain in the bath all night, and weighed in a weighing tube next morning, and their weights having been registered according to their numbers, stored away ready for use, as follows:

The milk to be examined is shaken, and with a pipette 5cc. are discharged into a small beaker 2 inches high by 1¼ diameter, of a capacity of about 30cc. weighing about 12 grams. This charged beaker is first weighed, and then a paper coil gently thrust into the milk very nearly to the bottom. In a few minutes the paper sucks up nearly the whole of the milk. The paper is then carefully withdrawn by the dry extremity of the coil and gently reversed, and stood, dry end downwards, on a clean sheet of glass. With a little dexterity all but the last fraction of a drop can be removed from the beaker and got on the paper. The beaker is again weighed, and the milk taken got by difference. It is of importance to take up the whole of the milk from the beaker, as I am disposed to consider the paper has a selective action, removing the watery constituents of the milk by preference over the fat.

The charged paper is next placed in the water oven on the glass plate milk-end upwards, and rough-dried. Mismanagement may possibly cause a drop to pass down through the coil onto the glass. This accident ought never to occur; but if it does, it is revealed in a moment by inspection of the surface of the glass, and the experiment is thereby lost.

In about an hour it is rough-dried and in a suitable condition for the extraction of the fat.

The method of Adams has been thoroughly tried by the English chemists, and has received the approval of the English Society of Public Analysts. It gives uniformly about .2 per cent. more fat in normal milk than the ordinary gravimetric methods.

In this laboratory we use the following modification of the process:

The blotting paper is replaced by thick filtering paper cut into strips 2 feet long and 2.5 in. wide. These are thoroughly extracted by ether or petroleum.

One end of the strip of paper being held horizontally by a clamp or by an assistant, 5cc. milk is run out by a pipette from a weighing bottle along the middle of the strip of filtering paper, being careful not to let the milk get too near the ends of the paper, and to secure an even distribution of it over the whole length of the slip. The pipette is replaced in the weighing bottle and the whole reweighed, and thus the quantity of milk taken is accurately determined. The strip of paper is now hung up over a sand bath in an inclosed space high enough to receive it where the air has a temperature of 100° C. (circa). In two or three minutes the paper is thoroughly dry. It is at once, while still hot, rolled into a coil and placed before cooling in the extraction apparatus already described.

The fat is dissolved by ether or petroleum, collected in a weighed flask, and, after thorough drying, weighed. I have already mentioned that by the use of asbestos paper I had hoped to be able to estimate the moisture in milk, but so far have not achieved the success which I believe is possible.

Of all the methods for the gravimetric determinations of fat in milk, I do not hesitate to say that the Adams method, properly carried out, is the best.

SOXHLET'S AREOMETRIC METHOD OF ESTIMATING FAT IN MILK.[1]

Caldwell and Parr[2] call attention to the difficulty which is often experienced in using Soxhlet's areometric method for the estimation of fat in milk. They say, speaking of the determination of fat by the lactobutyrometer: "But in this case, while the butyrometer gave tolerable results, Soxhlet's method failed entirely; even after standing five hours the layer of ether fat solution in the mixing bottle, which should be over a centimeter thick, was hardly a millimeter thick."

This experience is so much in harmony with my own that I thought it would be of interest to call attention to some of the difficulties encountered in working with Soxhlet's method.

Soxhlet's original paper was published in "Zeitschrift des Landwirthschaftlichen Vereins in Bayern," in 1880.

It rests upon the assumption that an alkaline milk shaken with ether will give all its fat to the ether, and this solution, being lighter than the rest of the mixture, will collect at the top, where it can be separated and its specific gravity determined.

The reagents used are ether saturated with water and a solution of caustic potash containing 400 grams to the litre.

The milk and reagents having been brought to a temperature of 17.5° C. are measured into a flask (I use an ordinary pint beer bottle), with pipettes furnished with the apparatus; 200cc. milk, 10cc. of potash solution, and 60cc. of the aqueous ether are the quantities to be employed. The milk is first placed in the flask and to this the potash solution added and shaken vigorously. Afterwards the ether is added and the shaking continued for one minute longer. The bottle is then put into water at a temperature of 17.5° C. and gently struck on the table in a vertical position at intervals of half a minute for fifteen or twenty minutes. At the end of this time the ether-fat solution has collected at the top, whence it is passed to the areometric cylinder by means of the rubber bulb blowing apparatus shown in figure 1.

Water is now added at a temperature of 16° C. to 18° C. to the outer cylinder, and after the temperature has become constant the density of the ethereal solution is read on the scale of the areometer. At the same time the temperature is read from the delicate thermometer attached

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[1] H. W. Wiley, Journal of Analytical Chemistry, vol. 1, no. 2.

[2] Am. Chem. Jour., vol. 7, p. 245.

to the areometer. The areometric degree is to be increased or diminished by the difference between the observed temperature and 17.5° C. as the former is above or below the latter. The percentage of fat is taken from a table which gives the numbers representing it for all degrees of the Soxhlet's scale between 43 and 66 for whole milk, or 2.07 to 5.12 per cent.; and for skimmed milk from 21.1 to 43, or from 0.00 to 2.07 per cent. It is thus seen that the scale includes all percentages of fat from nothing to 5.12. If a milk contain more than the latter percentage of fat it must be treated with a certain proportion of water before it can be examined by the Soxhlet's method.

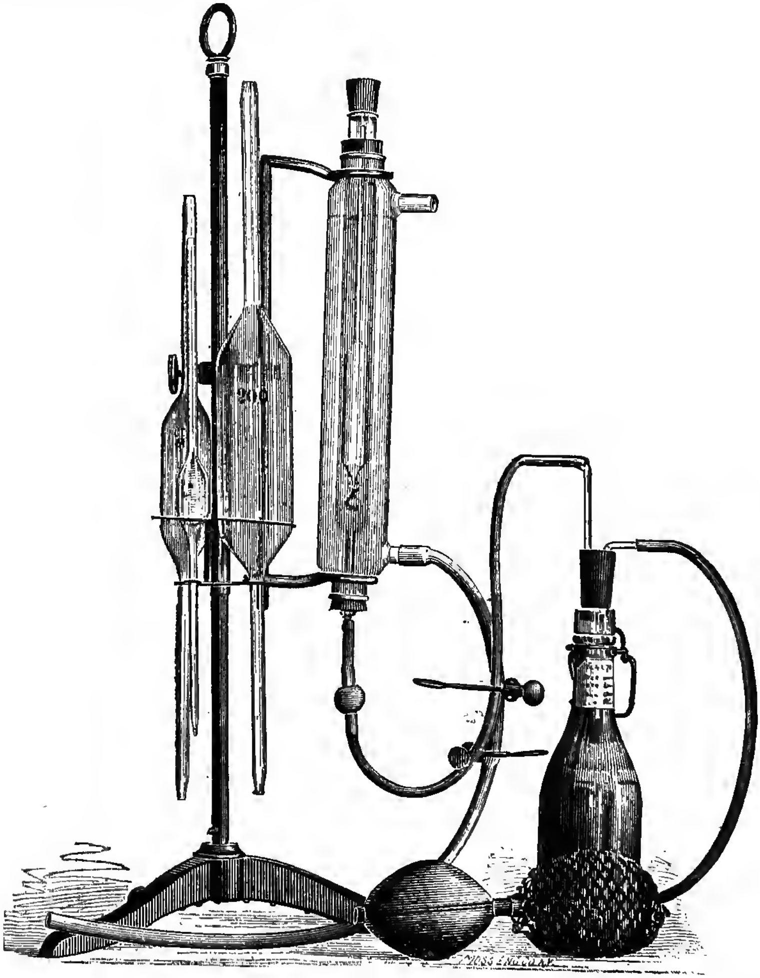

FIG. 1.

Both Soxhlet in his original paper and Liebermann[1] affirm that the ether retained in the form of an emulsion in the lower part of the liquid

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[1] Zeit. Anal. Chem., 1884, p. 478,

in the flask does not contain a trace of fat. It is, therefore, necessary to assume that the emulsion has always the same proportion of ether, otherwise there would be variations in the density of the clear supernatant solution. This may be entirely true with those milks which permit the ether solution to separate readily, but where the separation is difficult and a great deal of time is required for it to take place, it is possible that this assumption may not hold good.

Liebermann, who having once condemned Soxhlet's method, on account of the slow separation of the ether solution, introduced a modification of gently shaking the mixture and then pronounced it successful. He says[1]: "Therefore the manner of making the first shaking with ether is not immaterial. The shaking must not be violent (Soxhlet says 'schütteln eine halbe Minute kräftig),' but, nevertheless, always sufficiently so. The light vertical blows must also be skillfully applied. In fact, these are things which can only be attained by the skilled touch coming from experience."

It appears from the above, and my own experience fully convinces me of the truth of it, that in order to secure a proper separation of the ether fat solution, in all cases, one must be possessed of the art of legerdemain.

After giving the preference to his own volumetric method Liebermann ends his paper by the remark: "But if, on the contrary, the method of Soxhlet is employed, it will usually happen that a given milk must be examined twice, once to see whether the original method is applicable, and again, in case of failure, with the necessary modifications."

I think it will be readily agreed that such a method can find no place in the examination of the milk of individual cows for each sample of which a special modification of the method would have to be made.

Schmoeger[2] calls attention to the shortcomings of Soxhlet's method when milk poor in fat or skimmed milk is used. He says milk containing under 2 per cent. of fat must be evaporated with gypsum before it will give up its fat to ether. Yet it must not be forgotten that gypsum will give up something to ether, also. Schmoeger obtained from 20 grams of pure ignited gypsum 5mg. and from 20 grams sea sand 2mg. of extract. These facts must be taken into consideration when the gravimetric comparisons of fat per cents with the areometric results are made. Certain variations in the method of making these gravimetric determinations have lately come into use, which promise some improvement. Babcock[3] substitutes ignited asbestos for sand or gypsum. This, in my opinion, is a great improvement. Babcock[4] also dispenses with the evoprating dish and uses a tube open at both ends filled with as-

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[1] Zeit. Anal. Chem., *loc. cit.*

[2] Bericht ueber die Thatigkeit des Milchwirthschaftlichen Instituts zu Proskau, as quoted in Zeit. Anal. Chem., 1885, p. 130.

[3] Second Ann. Rept. Bd. Control N. Y. Exp. Sta., p. 167.

[4] *Loc. cit.*

bestos, with a plug of cotton at each end. The milk is absorbed by the asbestos, the tube is then placed in a steam bath and a slow current of air drawn through it by an aspirator. In two hours the desiccation is complete. The method gives satisfactory results. It is inconvenient, however, to work a large number of samples at once by this method. After drying, the tube is placed in a continuous extractor and the fat removed by ether. I have used the following modification of the gravimetric method. The evaporation is made in a schälchen half filled with fine, pure asbestos. About 5 grams of milk are taken for each determination. The water is driven off at 100° C. and after the total solids have been noted by weighing, the asbestos is removed to the extraction tube. The schälchen is then finely ground in a mortar, transferred to the extraction tube and the mortar and pestle thoroughly washed with ether. The extraction is then made in the usual way.

Adams[1] proposes the use of blotting paper rolled into a helical coil as the absorbent of the milk for the purpose of drying and extraction of the fat. Johnstone[2] modifies this method by using disks of blotting paper held in a circular platinum dish. Dr. Vieth[3] hesitates to approve the blotting paper method on account of the fact that blotting paper itself gives up a considerable extract to ether.

Allen and Chattway[4] recommend certain modifications in Adams' method, consisting in a peculiar method of winding the coil of blotting paper.

Thompson[5] proposes the use of filtering paper instead of blotting paper. I have had this method tried in the laboratory by Mr. Knorr and with pleasing results. Care must be taken, however, to use only filtering paper which has been previously thoroughly extracted. It was found that the filtering paper necessary to absorb 5 grams of milk was afforded by a piece 24 inches in length by 2.5 inches in breadth. This paper extracted with ether gave the following residues:

	Milligrams.
No. 1	11.4
No. 2	13.4
No. 3	12.0
No. 4	10.5
No. 6	12.2
No. 7	20.9
No. 8	15.5

Since the tables of the Soxhlet method are based on the old method of extraction they will have to be revised for the new data given by the Adams method.

One great advantage of the Adams method as modified by Thompson I have found to consist in the rapidity with which the preliminary dry-

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[1] Analyst, 1885, p. 48.

[2] *Ibid.*, 1885, p. 83.

[3] *Ibid.*, 1885, p. 86.

[4] *Ibid.*, 1886, p. 71.

[5] *Ibid.*, 1886, p. 73.

ing is accomplished. I have been able to put 5cc. of milk on a strip of paper, hang it over a sand bath and have it rolled and in the extractor within five minutes.

I mention this to show that even in the matter of gravimetric determinations by which the areometric method is finally judged, there is still a certain limit of variability.

I will return now to the subject more immediately under discussion. Schmoeger further says[1] that with skimmed milk, buttermilk, and such milks as have stood twenty-four hours on ice the ether-fat solution separates difficultly or not at all. To avoid this he recommends, after the addition of the potash, fully five minutes shaking, in order to form butter of the fat. Then the ether is added and the process continued as usual. In this case the percentage obtained by the areometric method must be increased .1 per cent. in order to agree with the gravimetric determinations. Schmoeger further recommends that skimmed milk or sweet buttermilk after treatment with potash be shaken with 10 grams potassium sulphate until the latter is dissolved. But this method also influences the specific gravity of the ether-fat solution, and the corrections to be made are found in the table given.[2]

Soxhlet himself[3] has called attention to the fact that with skimmed milk the ether-fat solution does not readily separate. A special scale has been constructed for such fat-poor milks giving areometric readings from 21.1 to 43, with the corresponding percentages of fat. For such milks Soxhlet proposes the following treatment, viz: A soap solution is made by taking 15 grams of a stearine candle, adding to it 25cc. alcohol, and 10cc. of the potash solution of the strength before given. The stearine is saponified by heating the mixture, and after the solution has become clear it is made up to 100cc. with water. From .4 to .5cc. of this solution is added to the milk under examination, and after a good shaking the rest of the process is carried on in the usual way. After the first thorough shaking on the addition of the ether the light jolting must be continued for fifteen minutes at intervals of half a minute in order to have the ether solution collect at the top. At longest, the solution separated after three or four hours.

Halenke and Möslinger[4] call attention to the fact that if samples of milk are kept for some time, even on ice, the ether-fat solution will no longer separate. They prefer in such cases a modification of Liebermann's method, which they describe. In general I may say the areometric method has met with the approval of all analysts who have used it with exception of Preusse[5], but Soxhlet[6] has shown that Preusse did not understand how to use the apparatus.

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[1] *Op. cit.*

[2] *Ibid.*, p. 132.

[3] Zeit. landw. Ver. Bayern, 1882, p. 18.

[4] Ver. Bay. Vertreter d Angewand. Chem., p. 110.

[5] Mittheil. Reichsgesundheitsamt, vol. I, p. 378.

[6] Zeit. landw. Ver. Bayern, 1881, p. 700.

The following chemists, in addition to those already mentioned, have given the method their entire approval: Egger, Kellner, Schrodt, Friedländer, Meissel, Fleischman, Hofmeister, Deitzell, Moser, Schreiner, Janke, Gerner, and Angström.

I will give now some of my own experiences with the areometric method:

The milk examined by me was mostly obtained from a neighboring dairy and was a mixture from forty cows. Samples were also bought from dealers in the city. The milk from the dairy mentioned was drawn at 5 p. m., and the examination made the following morning. This may partially account for the small success I had in securing a good separation of the ether-fat. The work extended from March 23 to May 7, 1886.

With the first series of samples in which the method of separation recommended by Soxhlet was followed ninety-three trials were made. In only four cases was the separation sufficiently good to get a reading within thirty minutes. A larger number of readings was obtained within an hour, and about half the number could be read at the end of three or four hours. Of the remainder about one-half could be read after twenty-four hours, and the rest did not separate at all. The results of reading the areometer at different times, however, showed that the density of the either-fat solution underwent quite a change. The following data will show the nature and extent of this change:

No.	Per cent. fat.		
	First results.	Results after 24 hours.	Difference.
1	4.16	4.08	−0.08
2	3.52	3.83	0.31
3	3.20	3.52	0.30
4	3.63	3.68	0.05
5	5.28	5.28	0.00
6	4.81	5.13	0.32
7	4.61	4.71	−0.10

From the above it is seen that there is no uniformity in the character of this change, but in the greater number of cases the areometer shows an increase in the percentage of fat on standing.

Attempts also to obtain a more perfect separation by varying the quantity of potash employed gave only conflicting results.

I was, therefore, forced to the conclusion that for general work Soxhlet's method would prove useless unless some method could be devised to secure a prompt and uniform separation of the ether-fat solution.

Various theories have been proposed to account for this peculiarity of milk in refusing to allow the ether solution to separate. Caldwell and Parr have supposed it to be due to the bran in the cow's food; Liebermann ascribes it to failure of manipulation; Schmoeger that it is caused

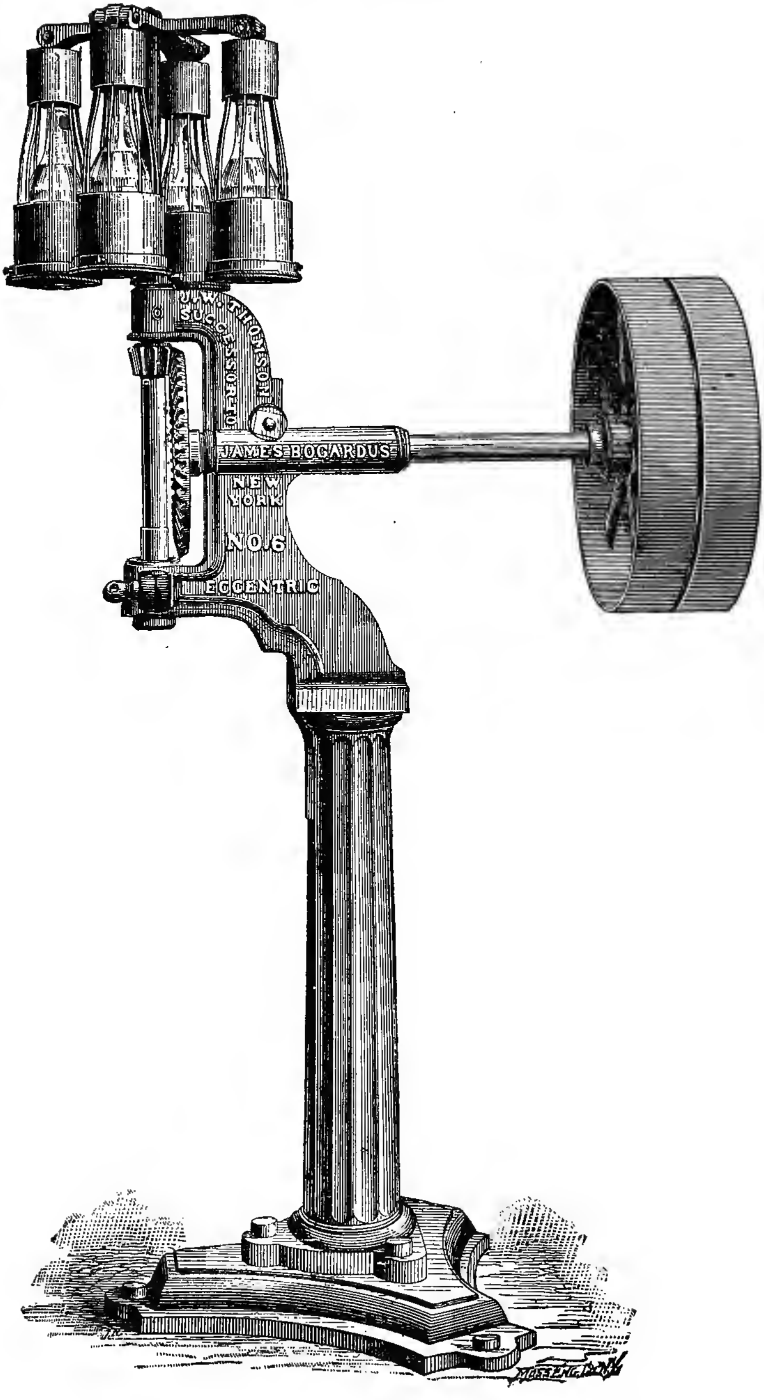
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FIG. 2.

by the milk standing on ice; Soxhlet thinks it is the result of deficiency of fat; and others attribute it to differences in age and breed of the cows. The résumé which precedes shows that not only the actual volume of the ethereal solution, but also the time of the separation required, has a serious disturbing influence on the specific gravity of the ether-fat solution.

Therefore, the method, in order to be of general application, must be subjected to some radical modification.

In this direction were the attempts to secure a more prompt separation by varying the amounts of caustic-potash solution employed. These attempts, as the record has shown, were entirely unsuccessful. Even if the different kinds of milk would permit a prompt separation by varying the quantities of alkali employed, the amount for each sample could only be determined by numerous and tedious experiments.

I, therefore, turned my attention in another direction. It seemed to me that a centrifugal machine might be used to secure this separation, and accordingly I had a castaway drug-mill, formerly used in the laboratory, modified so as to serve for this purpose. The machine was so arranged as to hold four separatory flasks and impart to them a high speed of rotation. The form of the machine, with modifications made, is shown in figure 2.

At this point of my investigations this apparatus was finished and I immediately subjected it to a trial.[1]

Four samples which had not separated at all at the end of three hours were placed in the apparatus and whirled for ten minutes. At the end of this time three of them had completely separated, and the fourth nearly so. The apparatus was set in motion again for five minutes, at the end of which time the separation of the fourth sample was accomplished.

The number of revolutions per minute of the machine was about 350.

It will be seen from the above that the very first trial of the machine was completely successful, securing a perfect separation of the ether-fat solution in a few moments in samples which previous trial, by the usual method, had failed to separate in several hours.

The next determinations were made on a sample of milk purchased at the Department restaurant.

Duplicate flasks were treated in the usual way to secure the separation, and only at the end of two and a half hours was enough clear solution obtained to get a reading: No. 1 gave 2.40 per cent. fat; No. 2, 2.30 per cent. fat.

The first set of samples of the same milk separated by the centrifugal gave the percentages following: No. 1, 5.52 per cent. fat; No. 2, 2.32 per cent. fat.

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[1] This apparatus was first described before the Chemical Society of Washington, May, 1886, and next at the Buffalo meeting of the A. A. A. S., August, 1886.

The separation took place perfectly in ten minutes, with a rate of revolution of about 300 per minute.

The second set of four samples was treated in the same way and separated completely in eight minutes. The following readings were obtained: No. 1 gave 2.36 per cent. fat; No. 2, 2.34 per cent. fat; No. 3, 2.31 per cent. fat; No. 4, 2.30 per cent. fat.

The third set of samples separated by the centrifugal showed the following percentages: No. 1 gave 2.23 per cent. fat; No. 2, 2.30 per cent. fat.

The volume of the clear ether-fat solution in each case was about 40cc.

The next trial was with milk also purchased in the Department restaurant. It proved to be one of the rare cases in which a reasonably prompt separation was secured by the old method. After thirty minutes about 25cc. of the ether solution had separated, which was enough to get a reading. Duplicate determinations were made: No. 1 gave 2.08 per cent. fat; No. 2, 2.04 per cent. fat.

Four separations of the same milk were also made with the centrifugal. Separation took place promptly in eight minutes at a speed of about 200 revolutions per minute, and the volume of ether-fat in each case was about 40cc.: No. 1 gave 2.01 per cent. fat; No. 2, 2.01 per cent. fat; No. 3, 2.00 per cent. fat; No. 4, 2.04 per cent. fat; which is an agreement as close as any one could expect.

Having thus shown that the centrifugal method was capable of making the areometric method applicable to almost every sample of milk, I undertook a new series of experiments. In all, 155 samples were subjected to treatment.

Of the 155 samples examined only 57 gave a good separation by the Soxhlet method in thirty minutes. Of the remaining 98, about half did not separate at all so as to permit a reading, and the other half only after several hours. Compare this with the centrifugal method, in which only 6 samples out of the whole lot required over fifteen minutes for separation and only one was abandoned as entirely inseparable, and the more general application of the process is at once apparent.

Of the 6 samples mentioned above, 3 were from the same cow, a grade Shorthorn, four years old, weight about 800 pounds, in milk since July 1, 1885. She gave 6 quarts of milk a day, was milked at 5 a. m. and 5 p. m. The samples of milk sent were taken at 5 p. m., on April 13, 17, and 22, respectively. The food received by this cow was the same as for all the others (36) from which samples were taken for analysis. They received at 5 a. m. 3 pounds of wheat bran, and the same of hominy chops, and then as much corn (maize) fodder as they could eat. The bran and chops were fed dry. In pleasant weather the cows were out until 3 p. m. They were then fed 10 pounds each of unthrashed oats. At 5 p. m. they got a half peck of chopped turnips and a repetition of the morning's feed of bran and chops.

The hominy chops used showed, on analysis, the following composition:

	Per cent.
Water	7.13
Ash	2.53
Ether extracts	9.03
Carbohydrates	69.32
Crude fiber	2.36
Albuminoids	9.63

Two of the other samples were received April 27 and 30 from a thoroughbred Jersey, four years old, weight about 600 pounds, in milk since July 1, 1885, giving at the time about 5 quarts daily. On the 29th of April samples of milk were also treated from the same cow, but after dilution the centrifugal separation, although more than usually difficult, did not require so long a time as on the occasion mentioned.

There is nothing shown by the analysis, by the breed of cow, nor by the food which gives any definite idea of the cause of the peculiarity in these milks which does not permit a speedy separation. It certainly is not the quantity of fat present, for other milks having the same, more or less, amounts of fat separated without difficulty. In the absence of any further evidence on this point we can only attribute the phenomenon to bovine idiosyncrasy.

In all 90 samples were compared by the usual method of separation and by the centrifugal. By the former method the mean percentage of fat obtained was 4.01 and by the latter 3.88. It thus appears that the numbers obtained by the centrifugal method must be increased by .13 in order to correspond to those of the old method. This discrepancy is readily explained when it is remembered that by the centrifugal motion the percentage of ether left in emulsion would naturally be less than with the former process of separation. The ether-fat solution thus becomes more dilute and consequently has a lower specific gravity. When, therefore, the percentage of fat in a milk determined areometrically, is calculated by the tables given for the old method of separation, it should be increased by .13 in order to represent the actual quantity present.

I think it safe to conclude from the data which have been obtained:

First, that the method of Soxhlet cannot be applied to the determination of fat in American milks, especially if they be from individual animals. It works somewhat better on mixed milks from a large dairy, but even in this case it is a rare thing to secure a prompt separation and in most cases the method would be very difficult of application.

Second, that by the use of the centrifugal machine described a prompt separation of the ether-fat solution can be obtained in all cases, even in those in which after forty-eight hours no separation whatever takes place by the usual method.

Third, that the estimation of the fat in milk by Soxhlet's areometer can only be accurately secured when standard volumes of aqueous ether and caustic potash are employed, when the volume of the ether-fat solution separated is sensibly constant and the time employed in separation sensibly the same. These conditions can only be secured by the use of the centrifugal machine described.

I propose to use a centrifugal apparatus also for assisting in the separation of the ether-fat solution in the lactobutyrometer; and it has already proved its usefulness in separating precipitates which subside very slowly.

I am of the opinion that such a machine would prove of great value in every chemical laboratory aside from its utility in determining the fats in milk.

Cronander[1] has proposed the following method of estimating the fat in milk:

A glass flask of 200 to 250cc. capacity, and two glass tubes constitute the chief parts of the apparatus. One of the tubes is furnished with a scale dividing it into ten equal parts. Below the last division the scale is expanded into a bulb, below which the tube extends for about 5cm. The other tube is bent to an obtuse angle and serves for the introduction of hot water into the flask to drive the fat into the measuring tube at the end of the operation. Both tubes are fastened to a cork stopper in such a manner as to have the measuring-tube end even with the under surface of the stopper, while the other extends almost to the bottom of the flask.

Of the milk to be analyzed 100cc. are taken at 17.5° C., 10cc. potash lye (200 grams to the litre) added and 30cc. aqueous ether. The flask is corked and thoroughly shaken. The ether-fat solution collects at the top (after one hour), and after evaporating the ether the residual fat is forced into the measuring tube by pouring water at 70° C. to 80° C. into the flask. The volume of the fat is thus determined and its per cent. can be calculated.

LIEBERMANN'S METHOD.[2]

This method, like that of Soxhlet's, depends on the separation of a fat from a mixture of milk and caustic potash by shaking it with ether.

Apparatus.—(1) A glass cylinder with ground glass-stopper, 26cm. high and 3.5cm. diameter. (2) Burettes of the form shown in Figs. 3, 4. (3) A glass flask holding from 45 to 47cc., according to size of burette employed; neck 1cm. diameter, with edge ground accurately in a horizontal plane. (4) Four pipettes, two of 50cc. and one each of 20cc. and 5cc. Before beginning the operation the flask is graduated as follows:

The burette, Fig. 3, is filled to the zero-point with pure water at the temperature of the working room. The water is now run out of the

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[1] Milchzeitung, vol. 11, pp. 161–164.

[2] Zeit. Anal. Chem., 1883, p. 383; 1884, p. 476; 1884, p. 87.

burette into the flask (previously carefully cleaned and dried), until the meniscus at the ground edge of the neck passes from the concave to the convex form, a change which is effected by a single drop. When the flask is nearly full at least five minutes should be allowed for the water to settle in the burette. The cubical contents of the flask are now noted from the burette.

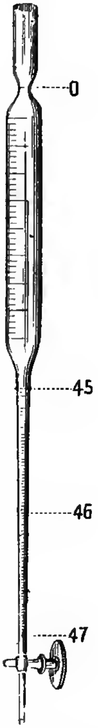

FIG. 3.

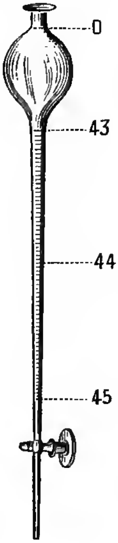

FIG. 4.

Reagents.—A solution of caustic potash having a specific gravity of 1.27. A solution of aqueous ether the same as is used in Soxhlet's method.

Manipulation.—Fifty cubic centimeters of the milk in the cylinder described above are treated with 5cc. of the potash solution, well shaken and allowed to stand for five minutes. This is next treated with 50cc. of the aqueous ether, and gently shaken for ten seconds. The cylinder is now allowed to stand for twenty minutes, receiving every half minute one or two light vertical blows. At the end of this time the separation of the clear ether layer is usually complete, yet it sometimes happens that some of the emulsion adheres to the under part of the flask in the form of a transparent covering. By means of a gentle rotating movement imparted to the cylinder this emulsion is collected and rapidly settles.

With a 20cc. pipette the clear ether solution is removed. Before this is allowed to flow into the flask that part of it which has been dipped into the solution is carefully wiped, so that no part of the potash liquid can drop into the flask. The ether solution is now evaporated, and the residue dried for at least half an hour at a temperature of 110° C., or what is still better, over a small flame until the odor of the decomposed butter is detected. The whole is now cooled in a dessiccator. It can now be weighed, if the weight of the flask is known, or it can be estimated volumetrically.

In case the estimation is made volumetrically, it must be carefully observed that the solidified fat contains no air bubbles. In case any air bubbles are noticed the fat must be again melted and warmed until they have all disappeared.

Into the flask containing the solidified butter fat water is again run out of the burette under the same conditions which obtained in determining the contents of the flask at first. It is of the greatest importance that all the measurements be made with the greatest exactitude, since a single drop too much or too little will influence the result.

In case some small particles of the butter fat are detached and swim about in the liquid no fear need be entertained that the results of the measurement will be influenced thereby.

It will appear at once that the difference between the volume of water originally held by the flask, and that which was necessary to fill it after the fat had been added, will represent the volume of the fat which was contained in the 20cc. of the ether solution. This number multiplied by 5 will give the volume per cent. of the fat at the temperature at which the experiment was made.

For converting volume per cent. into weight per cent. the following table is used:

Table for converting volume per cent. into weight per cent.

At temperature 15° C., volume per cent. ×	.91109=weight per cent.
16°	.90831
17°	.90642
18°	.90377
19°	.90170
20°	.90034
21°	.89897
22°	.89626
23°	.89216
24°	.88822
25°	.88703
26°	.87584
27°	.87463
28°	.87327
29°	.87191
30°	.87055

The following example will show the manner in which the above-described method of analysis may be reckoned.

The graduation of the flask shows a volume of 48cc. and 25cc. After the evaporation of the 20cc. of the ether solution 47.3cc. water was necessary to fill the flask. The volume of fat which was contained in the 20cc. of the ether solution is therefore 48.25—47.38=.95cc. This number multiplied by 5 gives 4.75cc. volume per cent. of fat. The temperature at which the estimation was made was 17° C.

Looking now in the table, opposite 17° C. we find the factor .90642 This number multiplied by 4.75 gives 4.3, which is equal to the per cent of fat by weight.

If it is wished to determine the per cent. by weight of butter per 100 grams, and not cubic centimeters as before, it is necessary to determine the specific gravity of the milk and to proceed according to the following formula:

$$P=\frac{p\ 1000}{S}$$

In this formula P denotes the number sought, p the quantity of fat found for 100 cubic centimeters, and S the specific gravity of the milk.

According to Wolff the percentage of fat obtained by the method just mentioned is too high, because a part of the ether used for separating the fat remains dissolved in the alkaline milk and this part of the ether contains no fat. The result is that the ethereal solution of the fat contains more of this substance than it otherwise would if the whole of the ether was separated.

Wolff has therefore proposed the following changes in the quantities of the reagents to be used and claims thereby to have obtained results which wholly agree with the estimations of fat by weight. For 50cc. of milk he proposes 3cc. of potash lye of 1.145 specific gravity and 54cc. of the aqueous ether.

Liebermann, however, in a review of the methods proposed by Wolff, maintains that his original method gives entirely reliable results.[1]

FLEISCHMAN AND MORGEN'S METHOD.[2]

Fleischman and Morgen describe a method of determining fat in milk when the specific gravity and total solids are known.

Fleischman[3] gives a more detailed study of this method.

The formulæ for the calculations are as follows:

$$(1)\qquad t=1.2f_{\wedge}/2{,}665\,\frac{100S-100}{S}$$

$$(2)\qquad f=0.833_{\wedge}/{-}2.22\,\frac{100S-100}{S}$$

In these formulæ t=per cent. total solids; f=per cent. fat in milk; S=specific gravity of the milk at 15° C.

The above formulæ may be simplified by putting

$$d=100S-100.$$

Tables are given to aid in the calculation of the results. By these formulæ when either the per cent. of fat or the total solids is known the other can be calculated with a high degree of accuracy.

ESTIMATION BY VOLUME OF CREAM.

The determination of the volume of cream gives a rough approximation of the percentage of fat in the milk.

The methods generally in use are based on the natural separation of the fat globules on standing and the estimation of the volume thereof in a graduated cylinder.

The creamometer of Chevalier[4] will serve as a type of all apparatus of this class. It is a cylinder 20cm. high and 40cm. diameter. The scale

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[1] Pharm. Centralh., vol. 24, p. 435; Zeit. Anal. Chem., 1884, p. 87.

[2] Jour. Landw., 1882, pp. 293–301.

[3] Jour. Landw., 1885, pp. 251 *et seq.*

[4] Becke, Milchprüfungs-Methoden, p. 40.

begins at 5cm. from the top of the cylinder and is extended downwards. Each mark is one-hundredth of the whole volume. Being filled with milk to the zero point and allowed to stand twenty-four to forty-eight hours, the percentage of cream is read directly on the scale.

Since the volume of cream formed depends on the shape of the vessel the temperature, and the time, this method is not reliable. This error is reduced to a minimum by the use of a centrifugal machine for separating the cream. A machine for this purpose has been constructed by Lefeldt.[1]

In 1883 I saw a very convenient machine which had been constructed, in the laboratory of the University of Illinois at Champaign. The apparatus already described for separating the ether fat solution in Soxhlet's method I have used with success in separating cream. The centrifugals used in separating the cream from the milk in large dairies are constructed on the same principle.

For a comparison of the numbers obtained by these processes with those given by the gravimetric determination, I refer to Becke's monograph.[2]

The Lactocrite.—This name is given to an instrument invented by De Laval[3] designed to separate the fat in milk after appropriate chemical treatment.

The test vessels used in this apparatus are cylindrical boxes made of silvered metal, with accurately ground hollow silvered stoppers. These stoppers are expanded at the bottom, and at the top are contracted, and end in a small hole. They are joined to a glass tube of small internal diameter. This tube is furnished with a jacket by which it can be screwed onto the stopper, and this jacket carries two longitudinal slits, through which the divisions in the glass tube can be read. In the bottom of the jacket is a hole, so that the glass tube and metal stopper form a canal open at both ends.

The centrifugal machine consists of a steel revolving disk. In the upper part of this there is a circular cavity, extending from which, like radii, are 12 holes to receive the test apparatus above described. These holes dip slightly downward. The disk is incased with a jacket having a removable cover, which prevents a too rapid fall of temperature during the operation. By means of appropriate apparatus the disk can be driven at the rate of about 6,000 revolutions per minute.

Preparation of the milk.—In an ordinary test tube put equal portions of the milk to be tested and a mixture of 20 parts of concentrated acetic and one part of sulphuric acid. The test tube is closed with a cork in which is fixed a glass tube, shaken, and heated for ten to fifteen minutes in a water bath with frequent shaking.

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[1] Becke, *op. cit.*, p. 43.

[2] *Op. cit.*, pp. 40–45.

[3] Ding. Poly. J., vol. 261, p. 219; Chem. Centralblatt, 1886, p. 798.

The cylindrical box above described is now filled from the test tube, the metal cork forced in, whereby the apparatus is entirely filled and the excess of milk forced out through the holes in the bottom of the jacket.

The disk having been warmed to 50° to 60° C. by hot water, is now filled with these samples and revolved for three to five minutes at the velocity already noted. The temperature of the disk should not be allowed to fall below 50° C.

At the end of this time the fat has completely separated and its volume can be read on the divisions of the glass tube. This division is so arranged as to represent .1 per cent.

Blyth[1] has made a comparison of the results furnished by the lactocrite with those obtained by Adams method. The results are given in the following table:

No.	Specific gravity.	Total solids.	Ash.	Fat.		
				Lactocrite.	Adams.	Difference.
1	1033.0	12.90	.80	3.40	3.44	—.04
2	1031.0	14.12	.80	4.55	4.69	—.14
3	1032.0	13.12	.77	3.60	3.57	.03
4	1030.5	13.07	.74	3.90	3.99	—.09
5	1032.0	12.98	.76	3.80	3.76	.04
6	1031.5	14.27	.76	4.90	4.84	.06
7	1032.5	13.84	.78	4.20	4.26	.06
8	1030.5	13.00	.76	3.70	3.69	.01
9	1031.0	13.51	.80	4.05	4.09	.04
10	1034.0	11.76	.76	2.10	2.07	.03
11	1035.0	9.99	.86	.45	.50	—.05

The table shows the greatest differences between the two estimations to be .14 and the mean difference to be .05 per cent.

It is estimated that with the lactocrite 48 determinations of fat in milk can be made in an hour.

A further discussion of the merits of the lactocrite is given by Faber.[2] He says:

> One great advantage of the lactocrite is the very simple way in which it is worked, so that no skill is necessary, but any dairyman may obtain as good results as the apparatus is able to yield. In order to illustrate this, I give below the results obtained by two persons at their first attempts; the first person is a dairyman used to heavy work. By way of a check I myself made some tests of the same milks:

By myself.	Dairyman.		
3.1	3.1	3.2	3.2
3.2	Failed	3.2	3.2
3.2	3.1	3.3	3.2
2.65	2.65	2.6	2.6
2.65	2.65	2.6	2.65

> These very favorable results are of importance as showing that in the lactocrite is at last found the long wished-for apparatus, possessing the two qualities not hither-

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[1] Analyst, 1887, p. 34.

[2] Analyst, 1887, pp. 6 *et seq.*

to combined—simplicity of construction and working and sufficient correctness for all practical purposes.

The lactocrite will, no doubt, be found invaluable for butter dairies, or dairy factories buying milk from different farmers, by enabling them to carry out the system of paying for the milk according to the amount of butter-fat which is the only fair system. At present, both in England and in other countries, the farmer whose milk will make butter at a rate of 3 pounds per 100 pounds of milk gets the same price as the farmers whose milk is so rich as to give 5 pounds of butter per 100 pounds of milk, which of course is most unfair. When milk is paid for according to the fat contained in it, the temptation to skim it is done away with, and, besides, a great encouragement is given to the production of rich milk.

The lactocrite will also prove of use for analysts who have access to a separator stand, as it will give in short time a more exact determination of the amount of fat than any other apparatus. In this connection it will be of interest to know that a special construction of it has been adapted to fit Dr. De Laval's small hand separator, worked by hand and requiring no foundation.

Sebelien has published a comparison of the results obtained with the lactocrite and Cronander's method with the gravimetric methods.[1]

Cronander's method gave in general results slightly below those furnished by the gravimetric and Soxhlet's processes.

Dr. Cronander, to avoid this error, has introduced a slight modification into his process by adding a little alcohol to the mixture of potash, ether and milk. The principle of the separation of the fat thus becomes the same as in the lacobutyrometer of M. Chevreul. By using this modified process it was found possible to bring the results up near to those of the gravimetric method.

The results furnished by the lactocrite showed an almost perfect agreement with the gravimetric numbers, the differences being usually within 0.05 per cent.

Attention must be paid to keeping the test tube holding the milk and acids well shaken, especially before pouring its contents into the metal box, and that the rest of the apparatus be pressed in the box at once when the milk has been found. In proceeding in this way no separation of the different parts of the test liquid is possible, and thus a fair average sample is recovered in the test glass.

Concerning the question of the advantages of the lactocrite as compared with other forms of apparatus for estimating the fat in milk Sebelien is somewhat conservative, but seems to think that the matter will soon be determined by comparative trials.

LACTOBUTYROMETRIC METHOD.[2]

This volumetric method depends on the separation of the fat from the milk by a mixture of ether and alcohol. The method has been carefully studied by Caldwell and Parr.[3]

A mixture of 75 parts of pure ether, 100 of absolute alcohol, and 135 of water are employed. The instrument employed is made of moderately thick-walled tubing (about

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[1] Landw. Versuchs-Stationen, vol. 33, pp. 393 *et seq.*

[2] Marchand, Instruction sur l'emploi du lactobutyrometer, Paris, 1856 and 1878.

[3] Am. Chem. Jour., vol 7, pp. 238 *et seq.*

1mm.); the stem is about 23cm., and the bulb about 8cm. long. It is important that the shoulder between the stem and the bulb should not be too abrupt. The bore of the stem is about 6mm., and it is graduated in $\frac{1}{10}$cc. The wider part of the tube has such a capacity that in passing from the lowest graduation on the stem to the inner end of the stopper in the lower mouth one passes from 5 to 33cc.; then the ether-fat solution will always come within the range of the graduation on the stem. This instrument differs from that originally given by Marchand only in being open at the bottom as well as at the top; this is a matter of some importance with reference to cleaning and drying it. The narrow stem in which the ether-fat solution collects makes more accurate readings possible than is the case with the wider tube with the same width of bore throughout, such as is now commonly used.

The manipulation is carried on as follows:

Closing the lower mouth with a good cork, 10cc. of the well-mixed sample of milk are delivered into the well-dried tube from a pipette, then 8cc. of ether (Squibbs's stronger) and 2cc. of 80 per cent. alcohol. Close the smaller mouth of the tube with a cork, and mix the liquids by thorough shaking, which, however, need not be either violent or prolonged. Both corks should be held in place by the fingers during this operation, and the upper one should be once or twice carefully removed to relieve the pressure within, otherwise it is liable to be forced out suddenly unless carefully watched, with consequent danger of loss of material. Lay the tube on its side for a few minutes and then shake it again, add 1cc. of ordinary ammonia diluted with about its volume of water, and mix as before by shaking; then add 10cc. of 80 per cent. alcohol, and mix again thoroughly by moderate shaking, and holding the tube from time to time in an inverted position while the lighter portion of the liquid rises to the surface.

Now put the tube in water kept at 40° to 45° C. till the ether-fat solution separates; this separation may be hastened by transferring the tube to cold water after it has stood in the warm water for a few minutes and then returning it to the warm water. Finally transfer the tube to water kept at about 20° C., and as the level of the liquid falls in the stem by the contraction of the main body of it in the bulb, gently tap the side of the tube below the ether-fat solution, to dislodge any flakes of solid matter that may adhere to the walls; then as this solution finally takes its permanent position in the tube, its volume will not be increased by the presence of such foreign matters. The readings are to be taken from the lowest part of the surface meniscus to the line of separation between the ether-fat solution and the liquid below it.

In this laboratory the use of the lactobutyrometer has been attended with the same difficulties, though to a less extent, which led to the modification of Soxhlet's method already noticed. The late improvements in both the volumetric and gravimetric determinations of fat in milk render a further discussion of the merits of the lactobutyrometer unnecessary.

OPTICAL METHODS OF ESTIMATING FAT IN MILK.

Since the white color of milk is due to the suspension of the fat globules, many devices have been contrived to determine the quantity of fat present by the opacity of the milk. The most convenient of these apparatus is the one designed by Feser.

It consists of a glass cylinder, in the lower part of which a smaller cylinder made of white glass is fixed. On this white glass are a few black lines. The outer cylinder carries a double scale, one set of numbers representing cubic centimeters and the other the percentage of fat.

Four cubic centimeters of milk are put in the cylinder and then water added until the black lines on the inner white cylinder become visible. The percentage of fat is then read from the top of the column of water in the large cylinder.

For a full description of the different forms of lactoscope the monograph of von der Becke may be consulted.[1] For sorting milks, the lactoscope in the hands of an experienced operator will give valuable indications in respect of the quantity of fat. A delicate lactometer, a good lactoscope, and an experienced operator will generally be able to determine whether a given sample of milk be whole or skimmed. The lactoscope, however, is of no value in determining with accuracy the percentage of fat present in a sample of milk.

ESTIMATION OF LACTOSE.

Chemical.—The chemical methods employed in estimating the sugar in milk will be fully discussed in another part of this bulletin devoted to the study of sugars and their adulterations.

Optical.—The optical method of determining the quantity of lactose in milk is both speedy and accurate when properly carried out. The principles which underlie this investigation and the proper method of carrying it out are given below.[2]

The usual method of determining milk sugar by evaporating the sample to dryness and extracting the sugar with alcohol after exhausting with ether requires a great deal of time and labor. If some reliable optical method could be devised the determination of the lactose in milk would be the work of only a few minutes. The difficulties which are encountered in seeking for such a method are numerous and serious, so much so that little credit has heretofore been given to any of the processes of optical analysis in use.

SPECIFIC ROTATORY POWER OF MILK SUGAR.

Crystallized milk sugar when first dissolved possesses a higher rotatory power than it has in the milk from which it was derived. This increased optical activity may be compared with the original by the ratio 8 : 5, nearly. After the solution has stood for twelve to twenty hours, or immediately on boiling it, this extra rotatory power is lost. In estimating the specific rotatory power of milk sugar the numbers given always refer to the constant and not the transient gyratory property.

Among the earliest numbers assigned to the rotation of lactose are those of Poggiale $(\alpha)_D = 54.2$ and Erdmann $(\alpha)_D = 51.5$ [Sucrose $(\alpha)_D = 66.5$]. Biot[3] places this number for lactose at 60.23, and Berthelot[4] at 59.3 for the transition tint $(\alpha)_j$. Hoppe-Seyler, in his "Handbuch der physiologisch-chemischen Analyse," gives this number at $(\alpha)_j = 58.2$. Since the

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[1] *Op. cit.*, pp. 45 *et seq.*

[2] Am. Chem. Jour., vol. 6, pp. 289 *et seq.*

[3] Compt. Rend., vol. 42, p. 349.

[4] Würtz Dict. de Chim., vol. 2, 1st part, p. 188.

ratio of $(\alpha)_D$ to $(\alpha)_j$ is 1 : 1.1306, the above numbers become for Biot $(\alpha)_D$= 53.27, for Berthelot $(\alpha)_D$=52.47, and Hoppe-Seyler $(\alpha)_D$=51.48. Hesse[1] observed the rotation number to be $(\alpha)_D$=52.67 when the solution contained 12 grams per 100cc. and the temperature was 15° C. On the other hand, when the concentration is only 2 grams per 100cc. the number assigned is $(\alpha)_D$=53.63. It appears from this that the specific rotation power of a solution of milk sugar diminishes with the increase of its concentration, and this view is adopted by Landolt, Tollens, and Schmidt.

The following general formula[2] is used to correct the reading of the polariscope for concentration of solution:

$$(\alpha)_D = 54.54 - .5575c + .05475c^2 - .001774c^3,$$

in which c = number grams sugar in 100cc. solution. These observations are contradicted by the work of Schmoeger,[3] who, in an elaborate series of experiments, using instruments of different construction and observing all necessary precautions, found the rotation number of lactose sensibly constant for all degrees of concentration up to the saturation point. In thirty-two series of investigations, in which the degree of concentration gradually increases from c=2.3554 to c=36.0776, and in which a constant temperature of 20° C. was maintained, the variations in the numbers obtained were always within the limits of error of observation. The mean of all these numbers fixes the value of $(\alpha)_D$ at 52.53.

According to Schmoeger variations in temperature have far more to do with changes in rotatory power than differences of concentration. The value of $(\alpha)_D$ falls as the temperature rises. Under 20° C. the disturbing influence of temperature is greater than above 20° C. At the latter degree $(\alpha)_D$ varies inversely about .075 for each 1° C. change of temperature. Pellet and Biard,[4] as a result of their observations, fix the rotatory power of milk sugar at 58.91 for $(\alpha)_j$ [δ. $(\alpha)_D$=52.12)].

After a careful review of the methods used in the above *résumé* and the numbers determined by them, I am inclined to accept the mean obtained by Schmoeger as the one entitled to the greatest credit. It also has the advantage of being almost the mean of all the various numbers which have been assigned as the specific rotating power of lactose, viz:

Poggiale	54.20
Erdmann	51.50
Biot	53.27
Berthelot	52.47
Hoppe-Seyler	51.48
Hesse	52.67
Hesse	53.63
Schmoeger	52.53
Pellet and Biard	52.12
Mean	52.65

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[1] Anal. Chem. u. Pharm., vol. 176, p. 98.

[2] Tucker, Sugar Analysis, p. 91.

[3] Ber. chem. Gessell., vol. 12, p. 1922 *et seq.*

[4] Bull. de l'Assoc. des Chimistes vol. 1, p. 171 *et seq.*

In the present state of our knowledge, therefore, the specific rotatory power of milk sugar should be taken at $(\alpha)_D = 52.5$. I propose, at an early date, to make a careful study of this subject, in order to fix, if possible, an exact number for the expression of the rotating power, and to examine the conflicting evidence respecting the influence of the degree of concentration on the same. The estimation of lactose in milk by the polariscope is rendered difficult also by the presence in milk of various albumens—all of which turn the plane of polarization to the left. As will be seen by the data given further along, the ordinary method of removing these albumens, viz, by a solution of basic lead acetate, is far from being perfect. If, therefore, a portion of the albumen be left in the liquid submitted to polarization, the rotation to the right will be diminished by its presence.

Hoppe-Seyler[1] assigns as the rotation power of egg albumen $(\alpha)_D = -35.5$, and for serum albumen $(\alpha)_D = -56$. Both acids and alkalies seem to increase the rotating power, which may with acetic acid reach $(\alpha)_D = -71$.

Fredericq[2] gives the rotation number for blood serum for the rabbit, cow, and horse at $(\alpha)_D = -57.3$, and for the dog at -44. Paraglobulin, according to the same author, has a rotation number $(\alpha)_D = -47.8$.

Milk albumen[3] has the following numbers assigned to it:

Dissolved in $Mg.SO_4$ sol.	$(\alpha)_D = -80$
Dissolved in dil. HCl.	$(\alpha)_D = -87$
Dissolved in dil. $NaOH$ sol.	$(\alpha)_D = -76$
Dissolved in strong KOH sol.	$(\alpha)_D = -91$

The hydrates of albumen[4] have rotation powers which vary from $(\alpha)_D = -71.40$ to $(\alpha)_D = -79.05$. From the chaotic state of knowledge concerning the specific rotating power of the various albumens, it is impossible to assign any number which will bear the test of criticism. For the purposes of this report, however, this number may be fixed at $(\alpha)_D = -70$ for the albumens which remain in solution in the liquids polarized for milk sugar.

The phenomenon of "birotation" in milk sugar has already been noticed. The problem of analysis of this sugar is, however, still further complicated by the facts pointed out by Schmoeger[5] and Erdmann,[6] that when milk is rapidly evaporated in a plain dish the sugar is left in the anhydrous state, and that this sugar in fresh solutions exhibits the phenomenon of "half rotation." When such sugar is extracted with alcohol and re-evaporated, it, doubtless, is still anhydrous. But in the calculation of results this sugar is generally estimated as containing water of crystallization, and thus an error, which Schmoeger reckons at as much as .2 per cent., is introduced into the results. This

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[1] Würtz, Dict. de Chimie, vol. 1, 1st part, p. 91.

[2] Compt. Rend., vol. 93, p. 465.

[3] Hoppe-Seyler in Handbook of the Polariscope, Landolt, p. 248.

[4] Kühne and Chittenden, Am. Chem. Jour. vol. 6, p. 45.

[5] Ber. chem. Gesell, vol. 12, 1915 *et seq.;* vol. 13, p. 212 *et seq.*

[6] Ber. chem. Gesell., vol. 12, p. 2180 *et seq.*

fact, not well recognized, combined with the knowledge that in the process of evaporation many particles of sugar must be occluded by the hardening caseine, tends to throw doubt upon the accuracy of estimating the sugar by the extraction method.

The work which I undertook had for its object the determination of the best method of preparing the milk-sugar solution for the polariscope, and a comparison of the numbers obtained by this instrument with those given by the ordinary process of extraction.

The reagents used for removing the albumens were:

(1) Saturated solution basic lead acetate, specific gravity 1.97.

(2) Nitric acid solution of mercuric nitrate diluted with an equal volume of water.

(3) Acetic acid, specific gravity 1.040, containing 29 per cent. $HC_2H_3O_2$.

(4) Nitric acid, specific gravity 1.197, containing 30 per cent. HNO_3.

(5) Sulphuric acid, specific gravity 1.255, containing 31 per cent. H_2SO_4.

(6) Saturated solution sodium chloride.

(7) Saturated solution magnesium sulphate.

(8) Solution of mercuric iodide in acetic acid; formula[1] KI, 33.2 grams $HgCl_2$, 13.5 grams. Strong $HC_2H_3O_2$, 20.0cc. Water, 64.0cc.

Alcohol, ether, and many solutions of mineral salts, hydrochloric, and other acids were also tried as precipitants for albumen, but none of them presented any advantages which would make a detailed account of the experiments of any interest.

Table No. 9 contains a record of the experiments which led to the adoption of 1cc. acetate of lead solution, or 1cc. acid mercuric nitrate, as the best amount of each for 50cc. of milk.

Nearly all the polarisations were made in a 400mm. tube. From two to four observations were made with each sample. An average of these readings was taken for each determination. In the calculations the value of $(a)_D$ was taken at 53 instead of 52.5, the number which subsequent investigations have led me to believe more exact. The instrument employed was a "Laurent Large Model" polariscope.

In all cases the volume of the solution was corrected for the volume of the precipitated caseine. The volume was assumed to occupy 2cc. for each 50cc. milk.

Since in the Laurent instrument the weight of sucrose in 100cc. to read even degrees on the scale is 16.19 grams $[(a)_D=66.67]$, it follows that the weight of lactose in 100cc. to read one degree on the scale for each percent. lactose present would be $16.19 : x = 53 : 66.67$; $x = 20.37$.

If 52.5 be taken as the value of $(a)_D$ for lactose, then $x = 20.56$.

In table No. 9, A indicates acetic acid, Pb basic acetate of lead, MR acid mercuric nitrate, &c. The letters C and H indicate the temperature; C denoting the ordinary temperature of the room, and H that the sample was heated to 100° C. and cooled before filtering.

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[1] Jour. de Pharm. et de Chim., vol. 10, p. 108.

The numbers obtained by extraction with alcohol are taken as the basis of comparison, not because I believe them to be more reliable, but because that method is the one generally employed in the estimation of milk sugar.

In the alcohol extraction the milk was evaporated to dryness in a thin glass capsule, the dish and dried residue pulverized in a mortar, washed with ether into a continuous extraction apparatus, exhausted with ether, and then with 80 per cent. alcohol for ten hours.

Duplicate analyses are indicated in the table by the small brackets.

TABLE No. 9.—*Percentage of milk sugar.*

Number.	Per cent. lactose extracted by alcohol.	Reagents employed in precipitating albumens.									
		Pb 1cc.	Pb 2cc.	Pb 3cc.	Pb 4cc.	Pb 5cc.	A 5cc.	Other reagents.			
		Per cent. lactose.									
1	4.57				(10cc.)	3.67	4.23				
2	4.52				2.45	3.57	4.14				
3	4.46	4.48					3.57				
4	3.92	4.19				3.35					
5	4.35		3.55				4.32				
6	3.71	4.01					3.00				
7	4.10	4.63 4.96 H					4.44 4.68 H				
8	4.16	4.29 4.33					3.80 3.67 H				
9	4.48	4.59 4.58 H					4.04 4.12 H				
								H_2SO_4			
10	4.10	4.12 H					3.47 H				
11	4.80	4.87 H			(4cc.)		4.44 H	4cc.	5cc.	6cc.	8cc.
12	4.77	5.02 H	4.82 H	4.50 H	4.31 H			4.70 H	4.76 H	4.76 H	4.78H
13	4.25	4.25	3.75	3.38	3.38			3.97	3cc 3.89		
								HNO_3			
14	4.22	4.90 H	4.58 H								
15	3.14	4.40 4.32 H						4.68 H	4.66 H	4.50 H	
16	3.30	4.43 H							3.98		
17	4.72	4.45	4.18	3.87	3.65	3.26		3.96	3.98 3.88 H	3.88	
18	4.88	4.87 4.87 H					4.37 4.25	(5cc.)	4.27 4.43 H_2SO_4		
19	4.31	4.71 4.86 H					4.43 4.43 H	4.51 4.79 H	4.59 4.59 H		
20	4.39	4.11									
21	4.70	4.17									
22	4.96	4.93 4.97 H					4.45 4.43 H		4.69 4.67 H		
23	4.60	4.41 4.45					3.86 3.86	3.90 3.90	3.94 4.10		
24	4.74	4.41 4.45 H					4.21 4.35 H	4.32 4.44 H	4.32 4.55 H		
25	4.59	4.33 4.37 H	NaCl	1cc. MR			3.94 3.93 H	4.03 4.10 H	3.98 4.10 H		
26	4.39		4.29 H	4.29 H							
27	4.60	4.18 H		4.66							
28	4.26	3.67 H		4.09							

Remarks on Table No. 9.—The results obtained by using various other reagents for the precipitation of the caseine, viz, $MgSO_4$, $CuSO_4$, HCl, &c., have not been entered in the table. In none of these cases was there sufficient encouragement to warrant an extended trial. In most cases the precipitation was slow or imperfect, and the filtration difficult.

One important fact should not be overlooked, viz, that any excess of basic plumbic acetate causes a rapid decrease in the rotatory power of the solution; whether this decrease is due to precipitation of the sugar or solution of the albumens does not clearly appear. Illustrations of this decrease are seen in analyses 2, 12, 13, and 17.

It seems to make little difference whether the precipatition is made hot or cold. The question of temperature is set forth in greater detail in the next table. From all the experiments made it clearly appeared that the best optical results are obtained by the use of a minimum quantity of basic lead acetate, or of either the acid mercuric nitrate or iodide. For 50cc. to 60cc. of milk, 1cc. of the lead acetate or mercuric nitrate solution of the strength noted, and 25cc. of the mercuric iodide solution are the proper quantities. It makes no difference, however, if a large excess of the two latter reagents is employed. Of the. three the last is to be preferred.

In Table No. 10 will be found the results of the comparative determinations of milk sugar by extraction with alcohol, by precipitation with 1cc. basic lead acetate, and the same with 1cc. acid mercuric nitrate, hot and cold, to each 60cc. of milk.

In many of the analyses the large differences in results by the three methods show a fault of manipulation, but all the results have been given without selection.

TABLE NO. 10.—*Percentage of milk sugar.*

No.	Reagents employed.					No.	Reagents employed.				
	Extracted by alcohol.	C. Pb 1cc.	H. Pb 1cc.	C. MR 1cc.	H. MR 1cc.		Extracted by alcohol.	C. Pb 1cc.	H. Pb 1cc.	C. MR 1cc.	H. MR 1cc.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*		*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
1	4.55	....	4.74	....	4.92	34	4.37	4.65	4.93	4.93	....
2	4.10	....	4.22	....	4.50	35	4.52	4.27	4.41	4.56	....
3	4.51	4.54	4.22	4.68	4.62	36	4.88	4.83	4.93	5.17	....
4	4.36	4.55	4.53	4.89	4.90	37	4.61	4.30	4.43	4.57	....
5	4.05	4.14	4.09	4.48	4.39	38	4.79	4.59	4.67	4.91	....
6	3.84	....	3.84	3.98	3.98	39	4.67	4.26	4.41	4.51	....
7	4.52	4.67	4.73	5.01	5.00	40	4.79	4.64	4.74	4.94	....
8	4.25	4.21	4.26	....	4.51	41	3.95	4.10	4.26	4.38	....
9	4.45	4.61	4.54	4.87	4.87	42	4.00	4.61	4.61	4.77	....
10	4.92	5.20	5.22	5.43	5.47	43	4.63	4.24	4.37	4.57	....
11	3.84	....	3.72	4.00	3.96	44	4.77	4.64	4.70	4.94	....
12	4.53	4.61	4.64	4.87	4.85	45	4.85	....	4.53	4.73	....
13	4.57	4.54	4.55	4.91	4.84	46	4.71	....	4.67	4.93	....
14	4.66	4.29	4.45	....	4.63	47	4.34	4.06	4.12	4.40	....
15	4.17	3.65	3.75	3.95	3.87	48	4.05	4.67	4.77	4.83	....
16	5.02	4.66	4.64	4.86	4.86	49	3.67	4.12	4.18	4.36	....
17	4.68	4.03	3.94	4.39	4.37	50	3.78	4.58	4.62	4.82	....
18	4.23	3.82	3.89	4.08	4.02	51	4.19	4.27	4.57	4.53	....
19	4.96	4.70	4.84	5.04	5.04	52	3.83	4.68	4.78	4.97	....
20	4.85	4.39	4.41	4.53	4.65	53	3.86	3.97	4.07	4.21	....
21	4.03	4.47	4.47	4.69	4.67	54	4.59	4.59	4.61	4.83	....
22	4.47	4.39	4.45	4.67	4.71	55	4.02	4.26	4.36	4.40	....
23	4.46	4.23	4.31	4.65	4.63	56	4.30	4.62	4.76	4.94	....
24	4.47	4.59	4.67	5.01	4.95	57	4.20	4.18	4.28	4.48	....
25	4.40	4.41	4.55	4.45	....	58	4.09	4.52	4.56	4.74	....
26	4.85	4.67	4.73	4.97	....	59	4.09	....	4.28	4.46	....
27	4.45	4.21	4.33	4.57	....	60	4.12	....	4.49	4.81	....
28	4.44	3.98	4.10	4.28	....	61	4.20	....	4.33	4.41	....
29	4.10	4.21	....	4.55	....	62	4.45	....	4.25	4.77	....
30	4.38	5.57	4.60	4.89	....	63	4.33	....	4.09	4.37	....
31	4.20	4.21	4.37	4.57	....	64	4.62	....	4.33	4.99	....
32	4.69	4.50	4.67	4.89	....						
33	4.52	4.27	4.41	4.41	....	Av	4.33	4.34	4.38	4.58	4.63

In the following table will be found the percentage of milk sugar obtained by using varying quantities of the mercuric iodide reagent, and a comparison of the results obtained with those given by the use of acid mercuric nitrate and basic plumbic acetate:

TABLE No. 11.—*Percentage of milk sugar.*

Number.	Reagents employed.					
	Pb.	MR.	Mercuric iodide.			
			20cc.	25cc.	30cc.	35cc.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
1	4.28	4.48	4.56	4.56	....	....
2	4.46	4.57	4.62	4 66	....	....
3	4.37	4.65	4.63	4.63	4.60	4.65
4	4.37	4.53	4.60	4.53	4.63	4.60
5	4.38	4.63	4.50	4.53	4.53	4.59
6	4.33	4.67	4.43	4.53	4.60	4.66
7	4.30	4.67	4.67	4.67	4.59	4.57
8	4.33	4.59	4.50	4.53	4.50	4.59
9	4.27	4.60	4.63	4.60	4.66	4.06
Av.	4.34	4.60	4.57	4.58	4.61	4.62

ALBUMEN REMAINING IN FILTRATE FROM LEAD ACETATE AND MERCURIC IODIDE SOLUTIONS.

From the fact that the polariscopic readings show that solutions of milk prepared with lead acetate have a lower rotating power than those prepared with mercury salts, it is to be inferred that the lead reagent either leaves certain soluble and transparent kinds of albumen in solution, or else dissolves a portion of those which are at first precipitated. To test the accuracy of this supposition a few analyses were made to determine the amount of albumen left in the filtrate from the lead and mercury reagents. At the same time different quantities of the mercuric iodide solution were used, in order to determine the amount which would give the best results. For 60cc. milk the quantity of mercuric iodide to be used should be 25cc. to 30cc.

In the following table will be found the percentages of albumen in the whey after precipitating with the reagents noted and filtering. Ten cubic centimeters of the filtrate were evaporated to dryness in a thin glass dish, and the dried residue (with the glass) burned with soda lime. The calculated nitrogen was then multiplied by 6.25 and the product taken as the percentage of albumen:

TABLE No. 12.—*Per cent. albumen in filtrate.*

From Pb.	From HgI_2. 15cc.	From HgI_2. 20cc.	From HgI_2. 25cc.	From HgI_2. 30cc.	From HgI_2. 35cc.
.0865	.1950	.0865	.0865	.0562	.0865
.1130	.0674	.0865	.0865	.0865	.0865
.1130	.0674	.0502	.1130	.0562	.0312
.0865	.0674	.0562	.1130	.0562	.0562
.1130	.0674	.0562	.0312	.0865	.0562
.1130	.0090	.0865	.0300	.0865	.1412
.1130	....	.1412	.1130	.1412	.0562
.1950	....	.1412	.1130	.1412	.1412
.1130	....	.1412	.0865	.1412	.0865
.1412	....	.1130	.1412	.1130	.0865
.1130	....	.1362	.0090	....	....
....	....	.1250	....	....	....
....	....	.0090	....	....	....
....	....	.0090	....	....	....
Av. . .1182	.0789	.0888	.0830	.0964	.0828

In Table No. 13 will be found percentages of albumen remaining in filtrate from lead acetate precipitation of forty-two samples taken from those represented in Table No. 10. From these two tables it is at once seen that the quantity of lævo-rotatory matter remaining in milk after treatment with basic lead acetate is much greater than in those samples treated with the two mercuric salts. This explains at once the higher per cent. of milk sugar obtained by using the last-named reagents, and shows that the use of lead acetate as a clarifying agent must be abandoned:

Table No. 13—*Per cent. albumen after precipitation by lead acetate.*

No.	Per cent.	No.	Per cent.	No.	Per cent.
1	.250	16	.237	31	.329
2	.306	17	.237	32	.305
3	.135	18	.169	33	.305
4	.272	19	.103	34	.237
5	.134	20	.271	35	.305
6	.239	21	.237	36	.339
7	.301	22	.271	37	.237
8	.305	23	.235	38	.374
9	.237	24	.271	39	.203
10	.330	25	.237	40	.373
11	.271	26	.237	41	.305
12	.305	27	.271	42	.339
13	.267	28	.339		
14	.237	29	.350	Av..	.278
15	.271	30	.374		

COMPARISON OF RESULTS OBTAINED BY EXTRACTION WITH ALCOHOL AND POLARIZATION.

By consulting Table No. 10, it will be seen that the percentage of sugar obtained by extraction with alcohol is practically the same as that got by polarization of the lead acetate filtrate.

Thus, the mean percentage of sugar by alcohol (65 analyses) is 4.32; by lead acetate, cold (53 analyses) is 4.34; by lead acetate, hot (64 analyses) is 4.38; by mercuric nitrate, cold (61 analyses) is 4.58; by mercuric nitrate, hot (24 analyses) is 4.63.

If now the milk sugar, as has already been intimated, exists in an anhydrous state after extraction with alcohol, the percentage of it after the addition of the molecule of water would be increased. Thus molecular weight of anhydrous milk sugar, 342: molecular weight of the hydrous $360 = 4.38 : x$, whence the value of $x = 4.61$. This agrees very nearly with the number obtained by acid mercuric nitrate.

By a study of Table No. 13 it is found that the mercuric iodide gives nearly the same rotatory power as mercuric nitrate, and also by combustion the filtrates from the milks clarified by lead acetate contain more albumen than those prepared with mercuric iodide. There is, therefore, every reason for believing that the numbers given by the mercury salts are nearer the truth than those from the lead.

It may be urged that the increased rotatory power observed by the mercury salts is due to the conversion by the dilute acids of a part of the lactose into galactose, which has a rotatory power greater than that of milk sugar. But when it is remembered that the quantity of acid introduced is extremely minute, that the samples need not be warmed, that they can be filtered and polarized within a few minutes of the time of the introduction of the reagents, the suggestion is seen to be of no force.

For example, in the acid mercuric nitrate it was found that the percentage of sugar was the same whether one, five, or ten cubic centimeters of the reagent were employed, and whether it was polarized immediately or after heating and cooling. It is evident that 1cc. of the reagent, containing less than a half cubic centimeter of nitric acid and diluted in 100cc. of liquid, could not exert any notable effect on the rotatory power of the solution.

In the mercuric iodide solution 20cc. of acetic acid are used for every 660cc. of the reagent.

Thirty cubic centimeters of this reagent contain, therefore, about 1cc. of acid. This in 100cc. of liquid, immediately filtered and polarized, could not affect in any marked degree the rotatory power.

Since combustion with soda-lime shows that the filtrate from the mercuric iodide sample is practically free from albumen, it is evident that the numbers obtained in this way must be a near approximation to the truth.

THE PROCESS OF ANALYSIS.

The reagents, apparatus, and manipulation necessary to give the most reliable results in milk sugar estimation are as follows:

Reagents.—(1) *Basic plumbic acetate*, specific gravity 1.97. Boil a saturated solution of sugar of lead with an excess of litharge, and make it of the strength indicated above. One cubic centimeter of this will precipitate the albumens in 50cc. to 60cc. of milk.

(2) *Acid mercuric nitrate*, dissolve mercury in double its weight of nitric acid, specific gravity 1.42. Add to the solution an equal volume of water. One cubic centimeter of this reagent is sufficient for the quantity of milk mentioned above. Larger quantities can be used without affecting the results of polarization.

(3) *Mercuric iodide with acetic acid* (composition already given).

Apparatus.—(1) Pipettes marked at 59.5cc., 60cc., and 60.5cc. (2) Sugar flasks marked at 102.4cc. (3) Filters, observation tubes, and polariscope. (4) Specific gravity spindle and cylinder. (5) Thermometers.

MANIPULATION.

(1) The room and milk should be kept at a constant temperature. It is not important that the temperature should be any given degree. The work can be carried on equally well at 15° C., 20° C., or 25° C. The slight variations in rotatory power within the above limits will not affect the result for analytical purposes. The temperature selected should be the one which is most easily kept constant.

(2) The specific gravity of milk is determined. For general work this is done by a delicate specific gravity spindle. Where greater accuracy is required use specific gravity flask.

(3) If the specific gravity be 1.026 or nearly so, measure out 60.5cc. into the sugar flask. Add 1cc. of mercuric nitrate solution or 30cc. mercuric iodide solution and fill to 102.4cc. mark. The precipitated albumen occupies a volume of about 2.4cc. Hence the milk solution is really 100cc. If the specific gravity is 1.030 use 60cc. of milk. If specific gravity is 1.034 use 59.5cc. of milk.

(4) Fill up to mark in 102.4cc. flask, shake well, filter, and polarize.

NOTES.

In the above method of analysis the specific rotatory power of milk sugar is taken at 52.5, and the weight of it in 100cc. solution to read 100 degrees in the cane sugar scale at 20.56 grams. This is for instruments requiring 16.19 grams sucrose to produce a rotation of 100 sugar degrees. It will be easy to calculate the number for milk sugar whatever instrument is employed.

Since the quality of milk taken is three times 20.56 grams, the polariscopic readings divided by 3 give at once the percentage of milk sugar when a 200mm. tube is used.

If a 400mm. tube is employed, divide reading by 6; if a 500mm. tube is used, divide by 7.5.

Since it requires but little more time, it is advisable to make the analysis in duplicate, and take four readings for each tube. By following this method gross errors of observation are detected and avoided.

By using a flask graduated at 102.4 for 60cc. no correction for volume of precipitated caseine need be made. In no case is it necessary to heat the sample before polarizing.

ESTIMATION OF THE ALBUMINOIDS.

The albuminoids in milk are most easily estimated by combustion with soda-lime or by previous conversion into ammonic sulphate and subsequent distillation from an alkaline liquid.

(1) *Combustion with soda-lime*—From 4 to 5 grams of milk measured from a weighing flask are evaporated to dryness in a schälchen either alone or with sand, gypsum, pumice-stone or asbestos. When dry the whole is rubbed up in a mortar, transferred to a combustion tube, and burned in the usual way. The nitrogen calculated from the ammonia formed multiplied by 6.25 gives the total albuminoids.

(2) The estimation of the albuminoids by Kjeldahl's method is so well understood that it will not be necessary to describe it here.

Following are the results of the analyses of milks made in this laboratory.

In table No. 14 are the results of the daily analyses of milk from the Maythorpe Dairy.

In table No. 15 are the numbers obtained with milks from various sources.

In these analyses the fat was estimated by the modified Soxhlet method, the sugar by the optical method, and the albuminoids by combustion with soda-lime.

TABLE NO. 14.—*Analyses of milk from Maythorpe Dairy.*

[All these samples were bought from D. M. Nesbit, College Station, Md.]

Number.	Specific gravity at 15° C.	Water.	Fat areometric.	Albuminoids.	Sugar.	Ash.	Total solids.
		Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
1	1.0348	87.31	....	2.57	4.60	.73	12.69
2	1.0340	86.96	....	2.81	5.00	.74	13.04
3	1.0333	87.62	3.64	2.61	4.80	.73	12.38
4	1.0339	....	4.12	2.84	4.85	.72	....
5	1.0326	87.69	4.00	2.72	5.05	.72	....
6	1.0316	87.08	3.92	2.63	5.14	.72	12.92
7	1.0337	87.27	....	2.80	5.19	.72	12.73
8	1.0362	87.98	3.21	2.77	5.10	.74	12.02
9	1.0341	80.97	3.66	2.75	5.19	.72	13.03
10	1.0333	87.62	3.98	2.78	5.27	.71	12.38
11	1.0315	87.89	3.51	2.80	4.99	.66	12.11
12	1.0367	87.54	4.25	2.80	4.99	.73	12.46
13	1.0334	86.09	4.62	2.98	5.02	.77	13.91
14	1.0368	85.44	......	3.92	4.65	.85	14.56
15	1.0328	86.73	....	3.04	5.00	.72	13.27
16	1.0358	84.58	4.73	4.05	4.67	.94	15.42
17	1.0335	87.84	4.98	3.15	4.92	.73	12.16
18	1.0325	87.33	....	2.63	5.04	.71	12.67
19	1.0323	86.60	....	2.03	4.95	.71	13.40
20	1.0353	80.24	....	4.13	4.70	1.21	13.76
21	1.0328	88.56	4.06	2.98	4.75	.55	11.44
22	1.0368	84.03	....	4.51	4.55	1.16	15.97
23	1.0328	84.64	5.22	2.84	4.90	.61	15.36
24	1.0325	88.10	4.24	2.73	5.04	.72	11.90
25	1.0329	86.17	....	2.76	4.99	.74	13.83
26	1.0349	85.46	4.74	3.64	4.95	.86	14.54
27	1.0323	80.59	....	2.87	4.82	.64	13.41
28	1.0369	84.93	....	3.61	4.38	.81	15.07
29	1.0330	85.73	4.92	2.98	5.14	.58	14.27
30	1.0344	86.60	....	2.59	5.17	.68	13.40
31	1.0339	86.85	....	3.18	6.00	.62	13.15
32	1.0363	85.67	....	3.92	4.70	.85	14.33
33	1.0324	86.55	....	2.80	4.84	.70	13.45
34	1.0369	83.75	....	4.73	3.69	.84	16.25
35	1.0339	85.66	....	3 29	5.13	.68	14.34
36	1.0346	....	5.08	3.11	5.02	.64	....
37	1.0350	85.67	4.87	2.94	5.67	.74	14.33
38	1.0340	....	4.75	3.22	5.02	.68	....
39	1.0340	87.15	4.47	2.76	6.30	.70	12.85
40	1.0355	88.98	4.78	2.76	5.57	.03	11.02
41	1.0325	87.71	4.00	2.69	5.02	.56	12.29
42	1.0333	87.04	4.11	3.04	4.75	.64	12.96
43	1.0338	85.49	5.25	2.84	4.90	.80	14.51
44	1.0318	86.92	4.26	2.87	4.80	.70	13.08
45	1.0343	86.10	4.66	2.69	4.89	.70	14.90
46	1.0343	87.18	3.75	2.59	5.03	.66	12.82
47	1.0328	87.44	3.57	2.66	5.01	.72	12.56
48	1.0334	86.79	4.03	2.80	4.80	.65	13.21
49	1.0342	85.85	4.81	3.15	4.97	.75	14.15
50	1.0327	87.87	4.18	2.80	4.80	.72	12.13
61	1.0338	80.56	4.02	2.87	5.19	.61	13.44
52	1.0319	86.10	4.96	2.63	5.20	.63	13.90
53	1.0319	87.63	3.38	2.76	4.95	.70	12.37
54	1.0318	87.50	3.51	2.66	4.49	.81	12.50
55	1.0343	85.37	4.72	3.15	4.85	.80	14.63
56	1.0323	86.85	3.75	2.87	4.77	.71	13.15
57	1.0338	86.14	4.08	2.87	5.07	.73	13.86
58	1.0332	86.23	4.50	2.66	5.15	.77	13.77
69	1.0317	87.82	3.16	2.56	4.85	.68	12.18
60	1.0325	86.51	4.21	2.66	4.85	.69	13.49
61	1.0354	84.64	....	3.43	5.15	.84	15.36
62	1.0339	86.89	4.00	2.84	5.02	.78	13.11
63	1.0340	86.98	3.94	2.80	6.23	.69	13.02
64	1.0340	87.74	3.72	2.69	5.27	.69	12.26
65	1.0305	87.96	3.13	2.66	4.87	.66	12.04
66	1.0331	87.57	3.88	2.63	4.95	.73	12.43
67	1.0305	89.20	3.57	2.76	4.33	.73	10.80
68	1.0328	89.01	3.26	2.87	4.99	.72	10.99
69	1.0314	86.32	3.02	2.73	4.67	.69	13.68
70	1.0320	87.22	4.65	2.39	5.27	.69	12.78
71	1.0320	86.89	4.53	2.98	4.97	.76	13.11
72	1.0334	86.55	4.11	2.87	5.15	.55	13.45
73	1.0317	88.35	3.55	2.73	4.67	.66	11.65
74	1.0345	87.12	3.59	2.91	5.10	.04	12.88
76	1.0310	88.01	3.66	2.69	4.49	.56	11.99
76	1.0340	87.62	3.67	2.45	5.40	.70	12.38

TABLE No. 14.—*Analyses of milk from Maythorpe Dairy*—Continued.

Number.	Specific gravity at 15° C.	Water.	Fat areometric.	Albuminoids.	Sugar.	Ash.	Total solids.
		Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
77	1.0336	86.16	4.75	2.84	5.13	.79	13.84
78	1.0320	86.80	4.36	2.45	5.15	.62	13.20
79	1.0320	84.73	4.25	2.56	4.90	.54	14.27
80	1.0328	86.34	3.50	2.91	4.89	.66	13.66
81	1.0318	88.71	3.71	2.59	4.80	.54	11.29
82	1.0334	86.94	3.97	2.31	5.49	.57	13.06
83	1.0325	86.01	4.58	2.91	5.13	.74	13.99
84	1.0336	86.41	4.23	2.63	5.22	.76	13.59
85	1.0311	87.73	3.61	2.73	4.60	.70	12.27
86	1.0332	87.09	3.79	2.69	4.99	.64	12.91
87	1.0321	86.54	4.11	2.63	4.90	.64	13.46
88	1.0322	86.73	4.41	2.24	5.30	.66	13.27
89	1.0333	85.75	3.85	3.01	5.25	.71	14.25
90	1.0312	88.66	4.64	2.34	5.23	.68	11.34
91	1.0328	87.87	3.61	2.48	5.25	.68	12.13
92	1.0341	86.94	4.06	2.38	5.49	.67	13.06
93	1.0322	87.63	3.75	2.56	5.02	.69	12.37
94	1.0322	86.59	3.78	2.28	5.29	.58	13.41
95	1.0312	88.30	2.73	2.45	4.93	.65	11.70
96	1.0322	87.42	4.30	2.45	5.25	.66	12.58
97	1.0332	87.65	3.68	2.41	5.27	.69	12.35
98	1.0332	87.22	4.35	2.66	5.37	.66	12.78
99	1.0313	88.93	4.79	2.59	4.95	.70	12.07
100	1.0328	87.29	4.07	2.38	5.37	.72	12.71
101	1.0325	88.31	3.05	2.45	5.50	.63	11.69
102	1.0345	87.40	3.65	2.41	5.45	.70	12.60
103	1.0345	87.78	3.60	2.38	5.33	.72	12.22
104	1.0355	88.38	3.97	2.63	5.75	.59	11.62
105	1.0330	88.24	3.14	2.63	5.22	.72	11.76
106	1.0345	88.60	3.73	2.45	5.67	.72	11.40
107	1.0335	88.58	3.06	2.63	5.30	.55	11.42
108	1.0317	88.86	3.57	2.24	5.05	.65	11.14
109	1.0325	87.89	2.61	2.45	5.07	.60	12.11
110	1.0320	88.47	4.02	2.34	5.23	.66	11.43
111	1.0320	88.97	4.68	2.52	5.27	.70	12.03
112	1.0315	87.85	4.67	2.34	5.07	68	13.15
113	1.0315	87.29	4.19	2.45	4.93	.66	12.71
114	1.0367	86.10	4.74	2.84	5.49	.69	13.90
115	1.0356	86.61	4.28	3.08	5.35	.76	13.39
116	1.0354	87.59	3.13	2.63	5.52	.73	12.41
117	1.0336	86.37	4.41	2.52	5.23	.73	13.63
118	1.0340	86.17	4.45	2.73	5.25	.74	13.83
119	1.0326	86.09	5.12	2.80	5.29	.69	13.91
120	1.0338	85.88	5.09	2.69	5.43	.72	14.12
121	1.0352	86.91	4.16	2.94	5.30	.74	13.09
122	1.0342	87.17	3.80	2.69	5.33	.70	12.83
123	1.0352	85.99	4.57	2.80	5.42	.71	14.01
124	1.0338	86.64	4.47	2.31	5.24	.72	13.36
125	1.0348	87.55	3.78	2.76	5.50	.79	12.45
126	1.0343	85.41	4.91	3.01	5.47	.71	14.59
127	1.0352	86.47	3.99	2.91	5.32	.72	13.43
128	1.0357	87.81	3.15	2.76	5.62	.77	12.19
129	1.0336	87.66	4.74	2.91	5.20	.72	12.34
130	1.0336	86.32	4.50	2.66	5.27	.71	13.68
131	1.0341	85.88	5.02	2.69	5.49	.69	14.12
132	1.0351	86.57	4.65	2.69	5.55	.75	13.43
133	1.0354	85.81	4.51	2.98	5.58	.76	14.19
134	1.0353	87.33	3.66	2.45	5.67	.89	12.67
135	1.0346	86.34	4.68	2.63	5.39	.75	13.66
137	1.0338	85.48	6.05	2.80	5.39	.79	14.52
138	1.0345	86.60	3.74	2.87	....	.77	13.40
139	1.0315	85.60	5.63	2.48	....	.71	14.40
140	1.0320	86.85	4.09	2.63	....	.73	13.15
141	1.0330	87.18	3.49	2.04	....	.73	12.82
142	1.0340	86.18	4.30	2.98	....	.74	13.82
143	1.0330	87.54	3.51	3.04	....	.60	12.46
144	1.0341	86.98	3.82	2.45	....	.67	13.02
145	1.0320	87.66	6.03	2.34	....	.74	12.34
146	1.0333	86.65	3.33	2.87	....	.66	13.35
147	1.0335	86.24	3.57	2.91	....	.58	13.76
148	1.0350	88.02	2.65	3.08	....	.73	11.98
149	1.0332	89.38	3.21	2.87	....	.59	....
150	1.0344	86.72	3.54	3.15	....	.78	13.28
151	1.0333	85.34	5.35	2.45	....	.70	14.66
152	1.0333	86.41	4.18	2.80	....	.60	13.59
153	1.0342	87.35	3.50	3.04	....	.75	12.65
154	1.0338	86.66	3.83	2.60	....	.64	13.34
155	1.0345	87.68	2.91	2.76	....	.59	12.32

TABLE NO. 14.—*Analyses of milk from Maythorpe Dairy*—Continued.

Number.	Specific gravity at 15° C.	Water.	Fat areometric.	Albuminoids.	Sugar.	Ash.	Total solids.
	.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
156	1.9353	86.43	3.87	2.37	....	.70	13.57
157	1.0320	86.46	5.29	2.52	....	.54	13.54
158	1.0335	86.99	3.91	2.63	....	.53	13.01
159	1.9349	86.89	3.73	3.19	....	.58	13.11
160	1.0339	86.73	3.07	2.91	....	.79	13.27
161	1.9333	87.59	3.30	2.66	....	.71	12.41
162	....	87.03	3.61	3.15	....	.60	12.97
163	....	87.73	3.43	2.66	....	.60	12.27
164	....	87.09	3.76	2.91	....	.64	12.91
165	....	....	4.04	2.73	....	....	....
166	....	....	5.41	2.27	....	....	....
167	....	....	3.76	3.11	....	....	....
168	....	....	....	3.85	....	....	....
169	....	....	3.94	3.92	....	....	....
Means ...	1.9334	86.95	4.08	2.78	5.05	.70	13.09

TABLE 15.—*Analyses of milk from various sources.*

1*	1.9315	88.14	4.73	2.88	4.67	.69	11.86
2*	1.0320	....	5.86	2.84	5.00	.71	....
3*	1.0316	87.48	5.05	2.84	4.75	.73	12.52
4†	1.0265	90.91	2.35	2.10	3.77	.62	9.09
5†	1.0254	91.59	2.01	1.96	3.63	.52	8.41
6†	1.9245	88.76	4.51	2.06	2.02	.56	11.24
7†	1.0285	89.84	2.68	2.17	4.23	.69	10.16
8‡	1.0335	86.97	4.07	2.91	4.92	.68	13.03
Means ...	1.9292	89.99	3.91	2.47	4.12	.65	9.54

* From C. J. Loomis, 1413 Stoughton Hill. † Department lunch room.
‡ Thompson's Dairy.

KOUMISS.

The use of koumiss, both as a beverage and in the sick-room, is rapidly increasing in this country, and for this reason I have thought it would be of interest to add here the results of the investigations on some home-made koumiss.[1]

Fermented mare's milk has long been a favorite beverage in the East, where it is known as "koumiss." Although the Tartars and other Asiatic tribes use mare's milk for the manufacture of koumiss, yet it is not the only kind that can be employed. Since the consumption of milk-wine has extended westward cow's milk is chiefly employed for making it both in Europe and America. Mare's milk is considered most suitable for fermentation because of the large percentage of milk-sugar which it contains.

König[2] gives as the average percentage of milk-sugar in mare's milk 5.31. The same author[3] gives as a mean of 377 analyses of cow's milk 4.81 per cent. of lactose. Dr. Stahlberg,[4] who brought forty mares from the steppes of Russia to Vienna for the purpose of using their milk for

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[1] Am. Chem. Jour., vol. 8, p. 200.

[2] Nahrungsmittel, p. 46.

[3] *Op. cit.*, p. 40.

[4] Tymowskis' Bedeutung des kumys, p. 12.

koumiss, found its percentage of lactose to be 7.26. On the other hand, ordinary mares that were kept at work gave a milk containing only 5.95 per cent. sugar. The quantity of milk-sugar in mare's milk is great, but there is a deficiency of fat and other solids. It appears to contain fully 89 per cent. water, while cow's milk does not have more than 87 per cent.

The process of manufacture is not uniform. In the East the mare's milk is placed in leathern vessels; to it is added a portion of a previous brewing, and also a little yeast. In thirty to forty-eight hours the process is complete. During this time the vessels are frequently shaken.

In the samples analyzed by me the milk was treated with a lactic ferment and yeast. After twenty-four to forty-eight hours' fermentation the koumiss was bottled. The bottles were kept in a cool place, not above 50° F., and in a horizontal position. When shipped to me they were packed in ice. After they were received in the laboratory they were kept on ice until analyzed.

METHOD OF ANALYSIS.

Carbonic dioxide.—The estimation of the carbonic dioxide was a problem of considerable difficulty. It was evidently impracticable to attempt opening the bottle and determining the gas in a portion of the contents. Fortunately I had access to a large balance which would turn with a milligram. On this I weighed the whole bottle, into the cork of which I had inserted a stop-cock such as is sometimes used with a champagne bottle. With the bottle of koumiss were also weighed two drying flasks containing concentrated sulphuric acid with their connections.

Having obtained the weight of the whole, the gas was allowed to escape slowly from the stop-cock and to bubble through the sulphuric acid in the washing flasks.

These flasks, previously to being weighed, were filled with the gas from an ordinary carbonic dioxide generator. After the gas had almost ceased to flow the bottle of koumiss was frequently shaken. It was also placed in a pail of water having a temperature of 30° C. After half an hour the gas ceased to come over.

The whole apparatus was again weighed. The loss of weight gave the quantity of free carbonic dioxide in the sample. After the analysis was completed the volume of the bottle was measured. It is fair to assume that at 30° C. the koumiss still contained an equal volume of dissolved CO_2. In determining the total CO_2 this volume, or its equivalent weight, was added to that obtained by direct determination.

By this method the CO_2 dissolved under pressure in the bottles is estimated separately from that which the koumiss contains in solution under the weight of one atmosphere. Since it is of no importance to separate the gas into these two portions, I have given it altogether in the tables, in volume, by weight; and in percentage by weight.

Acidity.—The samples examined showed under the microscope the acetic ferment, and a portion of the acidity was therefore due to acetic acid. It is the custom in giving the results of analyses of koumiss to represent the whole of the acidity as due to lactic acid. If ordinary yeast is used, and it generally is, it is possible that acetic acid may be formed. This appeared to be the case with the samples in question, since in distilling them a larger percentage of acid was found in the distillate than could have been expected had lactic acid only been present.

I made no attempt to separate these two acids, but estimated the total acidity, and then represented it in terms of both acids.

The direct titration of the lactic acid in the koumiss was attended with such difficulty that the attempt was abandoned. Whatever indicator was employed, the change in color was so obscured that no sharp reaction could be obtained.

To obviate this trouble the koumiss was mixed with an equal volume of saturated solution of magnesium sulphate. After shaking the mixture it was poured through a linen filter. The first portions running through were turbid. After refiltering these the filtrate was quite clear.

Better results were obtained by using with the koumiss equal volumes of alcohol. The filtrate from this mixture was uniformly bright. In this filtrate the acid was estimated by titration with standard sodic-hydrate solution, making the proper corrections for dilution and using phenol-phthalein as an indicator. I would recommend this alcoholic method of clarification to all who may have occasion to determine acid in milk.

Alcohol.—The alcohol was estimated by distilling 500cc. koumiss with 100cc. water until the distillate amounted to 500cc. This, being still turbid, was redistilled with a small quantity of water. The final distillate of 500cc. was used for the estimation of the alcohol in the usual way, viz, by taking its specific gravity and calculating the alcohol from tables.

Milk-sugar.—The milk-sugar was estimated by the method I recommended in a paper read at the Philadelphia meeting of the A. A. A. S.[1]

Fat.—Twenty grams of the koumiss were evaporated to dryness in a schälchen, the whole rubbed to a fine powder, and extracted with ether in a continuous extractor. The process of extraction lasted six hours.

Albuminoids.—The albuminoids were estimated by evaporating 5 grams of the material in a schälchen, rubbing to a fine powder with soda-lime, and burning with the same in the usual way.

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[1] Am. Chem. Jour., vol. 6, p. 289 *et seq.*

Water.—In a flat platinum dish partly filled with washed and dried sand 2 grams of material were weighed and dried to a constant weight at 100° C. Following are the results of the analyses:

TABLE No. 16.—*Analyses of koumiss.*

No. of analysis.	Weight of koumiss.	Volume of CO_2.	Weight of CO_2.	CO_2 by weight.	Acidity as acetic acid.	Acidity as lactic acid.	Alcohol.	Nitrogen.	Albuminoids.	Fat.	Watsr.	Milk sugar.
	Grams.	*Litres.*	*Grams.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*
1	747.415	2.543	5.009	.67	......	......	.87	.431	2.69	2.21	88.81	4.33
2	729.376	3.140	6.186	.85	.31	.47	.66	.412	2.58	2.15	89.53	4.31
3	768.575	3.179	6.269	.82	.34	.51	69	.483	3.02	2.07	89.15	4.33
4	736.035	3.281	6.463	.88	.30	.45	.81	.482	3.01	1.99	89.31	4.43
5	746.187	3.579	6.850	.91	.32	.48	.86	.423	2.64	1.67	89.97	4.43
6	750.247	2.973	5.757	.77	.27	.43	.70	.450	2.81	1.75	89.87	4.33
7	738.840	3.204	6.313	.85	.33	.40	.73	.402	2.89	2.44	89.01	4.48
8	752.550	3.203	6.428	.85	......	......	.77	.450	2.81	2.34	88.87	......
Mean	....	......	....	.83	.31	.47	.76	.449	2.56	2.08	89.32	4.38

It will be of interest to compare these results with those obtained by other analysts, both with koumiss from mare's milk and from other sources. As a mean of fourteen analyses of mare's milk koumiss, König[1] gives the following figures, viz:

	Per cent.
Alcohol	1.84
Lactic acid	0.91
Milk sugar	1.24
Albuminoids	1.97
Fat	1.26
Ash	0.30
Carbonic dioxide	0.952

The mean of two samples of koumiss made of cow's milk is given by the same author, as follows:

	Per cent.
Alcohol	2.64
Lactic acid	0.80
Milk sugar	3.10
Albuminoids	2.02
Ash	0.45
Carbonic dioxide	1.03

In nine analyses of koumiss[2] probably made of cow's milk the means are as follows:

	Per cent.
Alcohol	1.38
Lactic acid	0.82
Milk-sugar	3.95
Albuminoids	2.89
Fat	0.88
Ash	0.53
Carbonic dioxide	0.77

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[1] König, Nahrungsmittel, vol. 1, p. 68.

[2] *Op. cit.*, vol. 1, p. 68.

Interesting analyses of koumiss prepared from mare's milk have also been made by Dr. P. Vieth.[1]

The mares from which the milk was taken were on exhibition at the London International Exposition for 1884. These animals were obtained from the steppes of Southeastern Russia. The mares were from five to six years old, and were cared for and milked by natives of the country from which they were taken. When milked five times daily the best of these mares gave from four to five litres of milk. It is to be regretted that the milk-sugar, the most important ingredient of milk in respect of koumiss manufacture, was estimated by difference. Eleven analyses of the mixed milk gave the following numbers:

Table of analyses.

	Specific gravity.	Water.	Fat.	Albuminoids.	Milk sugar.	Ash.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Minimum	1.0335	89.74	0.87	1.71	6.30	0.26
Maximum	1.0360	90.41	1.25	2.11	6.82	0.36
Mean	1.0349	90.06	1.09	1.89	6.65	0.31

The koumiss from the above milk had the following composition:

Sample No.	Water.	Alcohol.	Fat.	Albuminoids.	Lactic acid.	Milk-sugar.	Ash.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	90.99	2.47	1.08	2.25	0.64	2.21	0.36
2	91.95	2.70	1.13	2.00	1.16	0.69	0.37
3	91.79	2.84	1.27	1.97	1.26	0.51	0.36
4	91.87	3.29	1.17	1.90	0.96	0.39	0.33
5	92.38	3.26	1.14	1.76	1.03	0.09	0.34
6	92.42	3.29	1.20	1.87	1.00	0.00	0.35
7	91.42	2.25	1.22	1.75	0.70	2.30	0.36
8	92.04	2.84	1.10	1.89	1.06	0.73	0.34
9	91.99	2.81	1.44	1.69	1.54	0.19	0.34
Mean	91.87	2.86	1.19	1.91	1.04	0.79	0.35

Collecting the above means together, we have the following comparative table:

Sample No.	Alcohol.	Lactic acid.	Sugar.	Albuminoids.	Fat.	CO_2.	Water.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	1.84	0.91	1.24	1.97	1.26	0.95	[2]92.47
2	2.64	0.80	3.10	2.02	0.85	1.03	88.72
3	1.38	0.82	3.95	2.89	0.88	0.77	[2]89.55
4	2.86	1.04	0.79	1.91	1.19	....	91.87
5	0.76	0.47	4.38	2.56	2.08	0.83	89.32

NOTES.—No. 1, mean of 14 analyses of koumiss from mare's milk; No. 2, mean of 2 analyses of koumiss from cow's milk; No. 3, mean of 9 analyses of koumiss, origin unknown, probably from skimmed cow's milk; No. 4, mean of 9 analyses of koumiss made from mare's milk, London Exposition of 1884; No. 5, mean of 8 analyses of koumiss from cow's milk, made by Division of Chemistry, United States Department of Agriculture.

[1] Landw. Versuchs-Stationen, vol. 31, pp. 353 *et seq.*

[2] By difference.

The comparison of the above results shows that the American koumiss differs from that of other countries in the following points, viz:

(*a*) The percentage of alcohol is quite low and as a consequence the percentage of sugar is high.

(*b*) American koumiss contains more fat; showing that it has been made from milk from which the cream had not been so carefully removed as in those milks from which the European koumiss was made. Mare's milk, as will be seen by the above analyses, contains much less fat and more sugar than that of the cow, thus making it more suitable for the production of koumiss. Good cow's milk, however, is suitable for the manufacture of koumiss after most of the cream has been removed. Should it be desired to make a koumiss richer in alcohol, some milk-sugar could be added.

The samples analyzed were kindly furnished me by Mr. Julius Haag, of Indianapolis. This koumiss makes a delightfully refreshing drink. When drawn from the bottle and poured a few times from glass to glass it becomes thick like whipped cream, and is then most palatable. It is much relished as a beverage, and is highly recommended by physicians in cases of imperfect nutrition. Those desiring to study the therapeutic action of koumiss should consult the monographs of Biel,[1] Stahlberg,[2] Landowski,[3] and Tymowski.[4]

CHEESE.

No studies of cheese have been made in this laboratory.

Caldwell[5] has given a résumé of the subject up to 1882, as follows:

Literature.—The subject of the adulteration of cheese receives only brief mention either in the journals or in monograph works on adulteration of food.

The Analyst[6] quotes from the Chicago Journal of Commerce the statement that soapstone, soda, and potash are added to cheese.

Hassell[7] states that cheese is adulterated with potatoes in Thuringia and in Saxony, and that bean meal is sometimes added in the place of potatoes; that Venetian red has been detected in several cases in the coloring of the rind, and as this color sometimes contains lead, and the rind is sometimes eaten, the fraud may be dangerous. He also says it is stated that blue vitriol and arsenic (green ?) are sometimes added, perhaps to give the appearance of age to the cheese, but he has never found them.

Ellsner[8] says that adulterations of cheese are not known. He mentions oleomargarine cheese as an article recently introduced in Germany. Griessmayer[9] also says that cheese is not adulterated; but he mentions in appropriate terms a practice of soaking certain kinds of cheese, such as Limburger, in urine in order to give

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[1] Untersuchungen über den Kumys und den Stoffwechsel während der Kumyscur.

[2] Kumys, seine physiologische und therapeutische Wirkung. St. Petersburg.

[3] Du kumys et de son rôle thérapeutique.

[4] Zur physiologischen und therapeutischen Bedeutung des Kumys. München.

[5] Second Ann. Rept. N. Y. S. Bd. of Health, p. 529.

[6] 1881, p. 29.

[7] Food and its adulterations, 1876.

[8] Die Praxis der Nahrungsmittel-Chemiker, 1880.

[9] Die Verfälschung der wichtigsten Nahrungs- und Genussmittel, 1880.

them in a short time the appearance of ripeness; such cheese can be made to show the reaction for murexide. He mentions the possible occurrence of poisonous metals, as copper, lead, or zinc in cheese, owing to carelessness in keeping it in metallic vessels or wrappings.

Fleischmann[1] quotes the results of Vogel's examination of cheese for lead; beyond 2 inches from the rind no lead was found, even in cases of cheese wrapped in very inferior tinfoil containing much lead; but in such cases lead was detected in the portions of the cheese immediately under the rind; 0.56 per cent. of lead was found in one instance in the rind of a cheese wrapped in tinfoil containing 15 per cent. of lead. Such cheeses are so little used in this country, however, that this matter has no general importance; but the information may serve as a warning to those who do eat them to be careful of eating the rind.

The same author mentions also the use of veratrin, sulphate of zinc, and arsenic to give to green cheese the strong biting flavor of old cheese, and the addition of blue vitriol to the milk in order to prevent huffing of the cheese.

Liebermann[2] mentions the danger in metallic wrappings, and states that verdigris is sometimes sprinkled over the cheese to give it the appearance of age. Blythe[3] states that washes containing arsenic and lead have often been applied to ward off flies, and as some people eat the rind, such practices may be dangerous.

Lard cheese.—About ten years ago dairymen were much concerned less the manufacture of cheese from skimmed milk and oleomargarine should seriously injure the reputation of American cheese abroad, and in that way hurt the dairyman's business here. Whatever chance this mode of making cheese may have had for success, it is now quite supplanted by the lard cheese which is made at over twenty factories in this State, under patents issued to H. O. Freeman in 1873 and to William Cooley in 1881.

In this process an emulsion of lard is made by bringing together in a "disintegrator" lard and skimmed milk, both previously heated to 140° Fahr. in steam-jacketed tanks; the "disintegrator" consists of a cylinder revolving within a cylindrical shell: the surface of the cylinder is covered with fine serrated projections, each one of which is a tooth with a sharp point; as this cylinder revolves, rapidly within its shell, the mixture of melted lard and hot skimmed milk is forced up in the narrow interspace, and the lard becomes very finely divided and most intimately mixed or "emulsionized" with the milk. This emulsion consists of from two to three parts of milk to one of lard; it can be made at one factory and taken to another to be used for cheese, but it is usually run at once into the cheese vat.

In making the cheese a quantity of this emulsion containing about 80 pounds of lard is added to 6,000 pounds of skimmed milk and about 600 pounds of buttermilk in the cheese vat, and the lard that does not remain incorporated with the milk or curd, usually about 10 pounds, is carefully skimmed off. These quantities of the materials yield 500 to 600 pounds of cheese, containing about 70 pounds of lard, or about 14 per cent.; about half of the fat removed in the skimming of the milk is replaced by lard (Munsell). It is claimed that no alkali or antiseptic is used, and that only the best kettle-rendered lard can be employed, because of the injurious effect of any inferior article on the quality of the cheese, and that before even this lard is used it is deodorized by blowing steam, under 80 pounds' pressure, through it for an hour.

According to many witnesses the imitation is excellent, for experts have been unable to pick out lard cheeses from a lot of these and full-cream cheeses of good quality together; and it may therefore be safely presumed that the general public would be quite unable to distinguish one from the other.

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[1] Das Molkereiwesen, 1879.

[2] Anleitung zur Chemischen Untersuchung auf dem Gebiete der Medicinpolizei, Hygiene und forensischen Praxis, 1877.

[3] **Food.**

The statistics of the manufacture of this kind of cheese as gathered from various sources, and partly by Inspector Munsell, are about as follows: Thirteen of the "disintegrators" are in operation, all in this State and none elsewhere. The production of cheese at the twenty-three factories engaged in the manufacture in this State during the six months ending November 1, 1881, was stated to amount to 800,000 pounds. None is made in other States, although it was stated before the Assembly Committee on Public Health,[1] in 1881, that it was made at the West. Before the same committee it was stated that some of the cheese was sold in New York City for consumption, but according to the best of my information, most if not all of it is exported. It is claimed that it brings from eight to ten cents a pound when full-cream cheeses sell at twelve cents, and "full-skim" cheeses at four or five cents; but New York dealers tell the inspector that the cheese brings but four cents a pound when its true character is known, and that it is for exportation only. The inspectors have not been able to find any cheese in the city markets which they had any reason to suppose to be lard cheese.

In two respects this kind of cheese can be considered as a fraud under the new food and drug law, unless sold under its distinctive name. It contains less fat, and fat of a cheaper kind, than the ordinary full-cream cheese contains, and, secondly, there are some grounds for the belief that the fat which is substituted for the butter fat is less wholesome than that. Rubner[2] in some investigations on the assimilation of various articles of food by the human subject found that lard was less digestible than butter; and the objection to oleomargarine butter on the ground of its indigestibility as compared with genuine butter may apply perhaps with more force to lard cheese; it only remains to determine by experiment whether the digestibility of the substances is increased by the operation of emulsionizing.

Skim cheese ("anti-huff cheese") is made, as is well known, from "full-skimmed" milk, without any attempt to replace the fat removed for butter. It is doubtful whether such cheeses are anywhere sold in a way to deceive consumers as to their character. To prove the quality of those cheeses, and especially to prevent them from puffing out, or "huffing," as it is technically called, from the abnormal generation of gases in the interior before they become fully ripe, patented "anti-mottling" and "anti-huffing" extracts are employed, consisting, it is claimed, only of caustic and carbonated alkali, saltpeter, and a little annatto, for coloring, dissolved in water. A qualitative analysis of one of these extracts by both Mr. Munsell and myself confirm this claim in one case; but another extract, said to be used at the West, was found to consist almost entirely of borax, which is a well-known antiseptic. The quantity of alkali and saltpeter said to be added to the cheese in this operation is small, in all less than five ounces to the milk and sour buttermilk for 100 pounds of cheese, and a portion of this must remain in solution in the whey; and there is no satisfactory evidence that such a quantity of borax as could be added to the cheese without affecting its taste would be prejudicial to the health for any ordinary quantity of cheese eaten. Gruber[3] shows that when this substance is taken into the system it seems to leave the organism very quickly and without affecting the system in any injurious manner.

As to the statistics of the manufacture of "anti-huff cheese," it is stated that in the most important section of this State for dairy products 4,500 cheeses of the best quality were made this year of skimmed milk and sour buttermilk with the aid of this extract. Before the assembly committee[4] it was affirmed that this cheese is consumed to

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[1] Fenner Committee. Testimony taken before Assembly Committee on Public Health in the matter of investigation into the subject of the manufacture and sale of oleomargarine-butter and lard-cheese. Hon. M. M. Fenner, chairman, 1881.

[2] Zeits. für Biologie, vol. 15, p. 115. Bied. Centralblatt, 1881, p. 394.

[3] Ber. Chem. Gesel., vol. 14, p. 2290. Zeits. für Biologie, vol. 16, p. 195.

[4] *Loc. cit.*

some extent in this country, but most of it is exported. It is claimed that nearly the same prices are obtained for the cheese as for full cream cheese, and that it is a good and wholesome article of food, containing nothing but what is found in other food. On the other hand, it is asserted that the excessive quantity of alkali supposed to be in the cheese makes it unwholesome, and that, like the lard cheese, it is a fraud on the public unless sold under a distinctive name; bringing nearly the prices of a full cream cheese, it is taken by consumers to be such. As to the first point there is no evidence *pro* or *con*, and the presumption is, as above stated, that there is no excessive quantity of alkali in the cheese. As to the latter point, it can be left to the interpretation of the law. Without question, a valuable constituent of the cheese has been removed and nothing of the same character has been substituted for it.

Water and fat determined were in small samples, each one of skim cheese made without anti-huffing extract, and with it. Both samples were taken with an ordinary cheese-tryer by Mr. Freeman, the patentee of the process, and sent through Mr. Munsell to me. The results of this partial analysis are given below:

	Water.	Fat.
	Pr. ct.	*Pr. ct.*
Anti-huff cheese	47.56	14.48
Ordinary skim cheese	47.00	16.77

There is nothing unusual in the composition of these samples as compared with skim cheese in general.

Poisonous cheese.—A sample of cheese, said to have produced sickness on the part of those who ate it, was sent to me by the Secretary of the Board, who received it from Inspector Smith. Cases of so-called "poisonous cheese" occasionally appear in different parts of this country and in other countries. Husemann,[1] quoted by Fleischmann,[2] mentions a number of instances from all parts of Germany, and also in England and Russia, resulting from eating old and especially sour milk and soft rennet cheeses. Scarcely ever is the result fatal, and recovery is rapid, because the vomiting, which is among the first symptoms, relieves the system of the dangerous matter. Voelcker[3] after noticing cases of sickness produced by cheese containing copper or zinc sulphate that had been added often surreptitiously by the dairymaid to prevent "heaving" of the cheese, gives an account of a case where all the cheese of a certain "make" sold in different places produced sickness. The cheese presented nothing abnormal in appearance, but his assistants, on eating less than a quarter of an ounce of it, were taken with violent vomiting and pain in the bowels, and a disagreeable mercurial after-taste was left in the mouth. No metallic poisons could be found in it, however, nor anything else abnormal except an apparently larger quantity than usual of fatty acids, giving a strong acid reaction to the cheese. He suggests that the poison is identical with the so-called sausage poison of German sausages made largely from coagulated blood, and says that a similar poison appears to be generated sometimes in pickled salmon, smoked sprats, pork or tainted meat, and that rancid butter may act as a poison. It disappears from the cheese when quite decayed. I have on previous occasions examined such cheese both chemically and with the microscope, without finding any cause for the physiological effect produced by it, or anything unusual. In one case the cheese was excellent in quality otherwise, but it nevertheless, when eaten to test the truth of the allegation against it, made me quite ill with the usual symptoms for a short time. In the present instance some of the cheese was offered to some kittens which are kept in stock for the use of the anatomical department of the University. At first only one would eat it and that one appeared to be quite sick the

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[1] Handb. d. Toxicologie, 1862.

[2] *Loc. cit.*

[3] Journ. Roy. Agric. Soc., vol. 23, p. 346.

next day. After her recovery the same kitten ate of the cheese again without any noticeable ill effects, and several others also ate of it without harm. It appears, therefore, that the illness of the first animal may as well have been caused by over-eating of rich food as by any supposed poisonous character belonging to the cheese.

I examined the cheese for poisonous metals in the rind, and for matters of the nature of alkaloids in the other part; no metals were found. With respect to the second test, a very small quantity of a substance, precipitated by alkali, soluble in ether, and giving with platinum chloride a yellowish flocculent precipitate, was obtained. These reactions indicate an alkaloid, but I have not been able as yet to carry the examination any further, and, moreover, it is not at all unlikely that a substance of the same character may be found in any ripened cheese as one of the normal products of the putrefaction. Therefore, this result obtained with the poisonous cheese can have no significance till normal cheese has been examined in the same manner without finding any evidence of the presence of alkaloids. For the present, therefore, we can only repeat what others have said who have given this matter their attention, that the cause of this peculiar property of cheese is probably an unknown organic substance, resulting from an abormal process of ripening.

Fickert[1] gives the results of some recent legal investigations on this subject. Cheese is so seldom the object of adulteration that when lately the daily papers stated that it was sometimes treated with urine in order to give it more quickly the desired odor and taste, it was considered as an isolated case. More worthy of note, therefore, was the discovery in a trial at Frankenberg, in Saxony, that mashed boiled potatoes had been used as an adulterant of cheese. This adulteration had already been discussed by Popperheim, and that, too, in cheese made especially for home consumption and not intended for commerce. This adulterant is easily detected by the microscope and by iodine.

Since the intrinsic value of cheese depends largely on its high content of albuminoids, viz, about 30 per cent., and since potatoes contain not much over a per cent. of these bodies, it is easily seen how greatly the value of the cheese is impaired by such an admixture.

TYROTOXICON.

The poisonous substance which sometimes is developed in cheese and milk has been isolated by Vaughn.[2] This substance, the chemical nature of which is not yet fully understood, has been found in cheese, milk, ice-cream, and oysters. For an account of its toxic properties consult Dr. Vaughn's papers.

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[1] Chem. Centralblatt, 1886, p. 956; Rep. d. ver. anal. Chem., vol. 6, p. 486.

[2] Paper read at Buffalo meeting A. A. A. S., Aug., 1886, Chem. News, 1886; Medical News, April 2, 1887, p. 369.

ADDENDA.

ESTIMATION OF FAT IN MILK.

Morse and Piggot[1] describe a method of estimating fat in milk by previous desiccation with dehydrated sulphate of copper. About 20 grams of the dried copper sulphate are placed in a porcelain mortar and 10^{cc} of milk added to it, being careful that none of the milk comes in contact with the mortar. The milk is dried in a very few moments, and the mass is then rubbed up with a little clean sand and transferred to an extraction tube. The mortar is then washed two or three times with from 10 to 15^{cc} of benzine, and the fat is extracted by treating in the extraction tube twelve times with the same quantity of benzine. The flask which has received the solution of butter is now placed on a water-bath and the volume of the solution reduced to 10^{cc} or less. The butter fat is now saponified with 20^{cc} of half normal solution of caustic potash. The excess of alkali is determined by a standard solution of hydrochloric acid. The results obtained by this method agree closely with the gravimetric determinations.

ESTIMATION OF WATER IN MILK.

The determination of water in milk is made in the following manner, described by F. G. Short[2]: About 2 grams of milk are placed in a Hofmeister capsule (schälchen) and dried at 110° C. in an oven containing a solution of chloride of calcium, boiling at 110° C.

When the fat is to be subsequently estimated the capsule is wrapped in a piece of prepared cheese-cloth, crushed between the fingers, and placed in an extraction tube.

SUBSTANCES SOMETIMES ADDED TO MILK TO MASK THE REMOVAL OF THE CREAM AND ADDITION OF WATER.

Where much water is added sugar is most frequently used to increase the specific gravity to the normal number.

Chalk, salt, annotto, turmeric, gum, dextrine, and cerebral matter have also been found in milk by Professor Weber.

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[1] Amer. Chem. Journ., vol. 9, p. 108.

[2] Amer. Chem. Journ., vol. 9, p. 100.

MILK ADULTERANT.

Felix Lengfeld, of San Francisco, has communicated to me the composition of a milk adulterant which has been largely used in San Francisco. The mixture consists of common salt, saltpeter, saleratus, a trace of caustic soda, and a large quantity of sugar. The color is imparted by caramel. These bodies are dissolved in an excess of water and the solution used for adulterating milk in any desired quantity.

OCCURRENCE OF ULTRAMARINE AS AN ADULTERANT IN MILK.

Thoms[1] has analyzed a milk which had a chalky appearance, and on standing showed an accumulation of a bluish liquid at the surface. Ultra-marine was found present in the proportion of 82.3 milligrams per liter.

FILLED CHEESE.

On the authority of the Produce Exchange Bulletin, I give the following account of the manufacture of filled cheese:

> The process consists in taking all the cream out of the milk by the separator, and then taking the skim milk up and charging the vat just before it is set with deodorized lard, cotton-seed oil, or other fat. The oil is taken up in the curd and mechanically held there, the cheese curd simply being used as a capsule in which to carry it. There is no assimilation or chemical affinity between the curd and its contents.

Prof. H. A. Weber, of Columbus, Ohio, has made comparative analyses of samples of genuine and artificial cheese, with the following results:

	Genuine cheese.	Artificial cheese.
	Per cent.	*Per cent.*
Water	35. 42	52. 73
Ash	2. 47	2. 69
Fat	34. 66	2. 63
Caseine, sugar, &c	30. 45	41. 95
Total	103. 00	100. 00

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[1] Pharm. Zeitung, vol. 32, p. 59.

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U. S. DEPARTMENT OF AGRICULTURE.
DIVISION OF CHEMISTRY.
BULLETIN No. 13.

FOODS
AND
FOOD ADULTERANTS.

BY DIRECTION OF

THE COMMISSIONER OF AGRICULTURE.

PART SECOND:
SPICES AND CONDIMENTS.

BY
CLIFFORD RICHARDSON.

WASHINGTON:
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PART 2.—SPICES AND CONDIMENTS.

U. S. Department of Agriculture,
Division of Chemistry,
Washington, D. C., March 15, 1887.

Sir: Having been directed by you to make an examination of the adulteration of spices and condiments, I have been engaged at different times during the past year upon the subject, and with some aid from the assistants in the laboratory in performing the chemical determinations have prepared the following report, which forms part 2 of Bulletin No. 13 of this Division. It has been my endeavor to collect all the available information in regard to the extent and character of this adulteration in all parts of the world, and to describe for the benefit of investigators the means to be employed for its detection. More particularly I have entered into an examination of the spices and condiments found in the markets of Washington and Balitimore, and have tested with them the methods of investigation which are described.

It has seemed appropriate to divide the report into two parts, the first being devoted to a popular exposition of the origin of the spices and the means employed for their adulteration, while the second contains a more extended technical discussion of the subject and of the results of analysis, both microscopical and chemical, and the methods which have been proposed by recent authorities for the detection of sophistication. I have been assisted in the chemical work by Messrs. Knorr, Trescot, and Fake, assistants in the laboratory of the Division, who have completed a large portion of the determinations under my supervision, and I am indebted to Dr. Battershall, of New York, for the privilege of examining the advanced sheets of his work on food adulteration.

To the standard authorities, Hassall, Koenig, Schimper, and others, I owe much for the information derived from them, although their work is not entirely applicable in this country, and I have copied from Schimper some of his diagramatic drawings of the spices, which, although extremely deceptive to the beginner, may in their proper way be made of value for reference. Other sources of information I have acknowledged in the body of the report.

The amount of adulteration which has been detected is extremely large and of a nature which apparently arouses but little prejudice on the part of the consumer, being free from associations of uncleanliness. Could only a portion of the unfortunate dislike for oleomargarine be directed toward the spices, the result would be that much wasted energy would be turned into a profitable channel. The necessity for some means for the suppression of the present universal sophistication of spices and condiments seems urgent.

Very respectfully,

CLIFFORD RICHARDSON.

Dr. H. W. WILEY.

SPICES AND CONDIMENTS.

PART I.

Under this head are included substances which while not in themselves foods serve to render the latter more palatable, and to stimulate digestion. They occupy an important position in the diet of the human race, and are largely subject to adulteration or sophistication. Among the more common are: Peppers of various kinds, mustard, cloves, cinnamon, cassia, allspice or pimento, nutmeg, mace, ginger.

These substances are so often and seriously adulterated because it is readily accomplished, owing to the custom of putting them on the market in a ground condition, which prevents the recognition of quality from mere appearance, and because so many cheap substitutes or diluents are readily found which resemble the real article. In addition, the demand on the part of the poorer classes for the cheapest possible supply and the competition brought about by this demand in the trade has, owing to general high prices attached to most of the spices of good quality, fostered and extended the practice.

At the present time in several of our largest cities the price to be paid for a spice is named by the retail dealer, and he is then furnished from the spice-mill with a mixture containing the largest amount of pure material which can be supplied for the money, the necessary weight being made up of diluents of some cheap but harmless substance, different grades being distinguished by one firm as pure, extra, No. 1, and superior, none of which are pure and many of which are mere variation in labels with none in quality. As examples, the fact that a New York firm, it is understood, in a short time used and put upon the market in their spices more than 5,000 pounds of cocoanut shells, and the following quotations from a journal devoted to spice milling show how universal and open the custom has become. On a prominent page is found:

"All necessary information for spice manufacturing supplied."

Attention should be paid to the use of the word manufacturing.

Then the following advertisements appear, from which the names have been stricken out:

—— ——,

MANUFACTURER OF

SPICES, SPICE MIXTURES, AND MUSTARDS,

181 —— *Street, N. Y.*

Goods made to order for Wholesale Grocers and Druggists; also, Grinding done for Jobbers who pack their own Goods.

Spice Mixtures and Cayenne Pepper a Specialty.

—— ——,

17 —— STREET, NEW YORK,

MANUFACTURER OF

ALL KINDS OF SPICE MIXTURES.

My celebrated brand of P. D. Pepper is superior to any made.

Samples sent on application. Goods shipped to all parts of the United States. Spice mixtures a specialty. Spices ground for the trade. We are the Inventors of Suction Coolers.

The firms advertising do not hesitate to call themselves manufacturers instead ot millers.

It is easy to see how difficult it must be to bring this state of things to an end without some governmental action, it being improbable that by any means of agreement among themselves the grinders of spices could unite in doing away with the practice, or that any education of the masses will teach them to refuse to purchase a ground spice at a price which is far below that of the unground article. This alone, the relation between the prices of ground and unground spices, is often sufficient to point out the fact that a ground spice must be largely diluted, and, on the other hand, when purchasing from a reliable dealer, a slight increase in cost over that of the spice in its original form is fair evidence of the purity of the powder. Those who desire pure ground spices can almost always obtain them by paying their value. They are by no means uncommon in the market, but as long as there are those who do not know that it is for their interest to buy the best, rather than a cheap article on account of its low price, such people must suffer or be protected by legal enactments which shall prevent and prohibit the existence of such mixtures. Until this is done the supply of a demand which certainly exists may be considered to be, at the least, justifiable on the part of the spice millers, and education of those ignorant of the state of the trade must be the preliminary to legislation upon the subject. When proper legislation has found a place on the statute-books the manufacturers will find themselves in a position where, without detriment to themselves, they can all unite in giving up the practice. Under the laws for the prevention of the adulteration of foods which have been in operation in Germany, England, France, Canada, and a few of our States, during a longer or shorter period of time, a large share of attention has been given to the adulteration of spices and condiments and the means of detecting them. Fortunately the latter are not difficult, and the results have been an awakening of the communities in these countries to an appreciation of the advantages of pure spices and the placing of the method of detection on a more certain basis.

EXPERIENCE IN COUNTRIES HAVING PUBLIC ANALYSTS.

In England the public mind had been so far educated by the publications of private investigators, such as Hassall, that in 1860 laws were passed for the prevention of the adulteration of food and drink. These have been modified and repealed, so that the present law dates from 1875 and amendments of 1879. Unfortunately there is no Government report, of which we are aware, upon the results of the scientific work done by those employed under the act, and we are indebted to the Society of Public Analysts for a large portion of the information which is at our disposal in regard to adulteration in England. We have also in the publications of Hassal, Blyth, and Allen volumes which give the most recent scientific data as to the best methods for the detection of adulteration, and illustrations of the forms in which foreign matter occurs. On Dr. Hassall's work is founded many of our present methods of examining foods microscopically, and especially spices and condiments. In the Analyst, the publication of the Society of Public Analysts, will be found among the proceedings of the society, in papers of individuals, and in reports of prosecutions, much information in regard to the status of adulteration in England during the last eleven years, including the material used for adulteration of spices and the means of detecting it. The lack of an official publication of the results of all that has been done in regard to particular samples and of the methods employed for their examination is, however, much to be regretted. There is the same difficulty in Germany. The law of the Empire of 1881 provides for the prevention of the adulteration of the substances which we have under consideration, but no reports on the execution of the law or of the results, scientific or otherwise, have been made available to us. Much, however, has been published in the German technical and scientific journals on the methods of detecting adulterants, which is of the greatest value.

In France, the laboratory of the prefecture of police of Paris, which has control of the investigation of the food supplies of that city, makes an elaborate report annually, of which, however, but a small portion is devoted to spices, although they are recognized as being largely adulterated, pepper, for example, being mixed to an astonishing extent with ground olive stones. Other cities of France have municipal laboratories whose reports, if any there are, have not reached us.

In this country Canada makes a much better statement of the results which have followed the enforcement of the adulteration-of-food act of 1876 than is done anywhere else. The commissioner of inland revenue has published annually for eight or ten years a statement showing the entire number of samples analyzed, the persons supplying them, and their composition and adulterants. Spices occupy a prominent position in the reports, and a collation of the results of the investigations of the several public analysts is of interest. In 1878, when the reports first become available to us for reference, the summary of the spices analyzed showed:

Articles.	Unadulterated.	Adulterated.	Per cent. adulterated.
Allspice	1	11	92.5
Cassia	2	2	50.0
Cloves	3	15	83.3
Cinnamon	3	6	66.6
Ginger	5	8	61.5
Mustard	....	38	100.0
Pepper	12	28	70.0

This enormous amount of adulteration, amounting to nearly a universal custom, was followed in 1879 by a similar report:

Articles.	Unadulterated.	Adulterated.	Per cent. adulterated.
Allspice	6	10	62.5
Cassia	....	1	....
Cloves	7	9	56.2
Cinnamon	3	16	84.2
Ginger	5	6	55.5
Mace	5	1	16.6
Mustard	3	....	....
Pepper	22	21	48.8

In 1880 there were reported as the results of the public analysts' work:

Articles.	Unadulterated.	Adulterated.	Per cent. adulterated.
Allspice	15	6	31.6
Cloves	12	10	45.5
Cinnamon	6	16	72.7
Ginger	9	8	47.1
Mustard	....	16	100.0
Pepper	24	18	42.9

There is a very slight improvement apparent, but it must be remembered that in examinations of this description the specimens selected are always of a suspicious nature, those which are already known to be pure being omitted, so that year by year the list of brands which are excluded from examination increases.

In 1881 and the following years results were tabulated as follows:

Articles.	1881.		1882.		1883.		1884.		1885.	
	Genuine.	Adulterated.	Genuine.	Adulterated.	Genuine.	Adulterated.	Genuine.	Adulterated.	Genuine.	Adulterated.
Allspice	8	17	......	....	......	......	......	......	......	.....
Cloves	6	7	......	......	......	......	......	......	12	10
Cinnamon or cassia	12	12	.....	......	......	......	......	......	......	......
Ginger	7	5	7	16	......	......	......	......	37	29
Mace	1	1	......	......	......	......	......	......	......	......
Mustard	2	2	......	......	......	......	......	......	11	*39
Nutmeg	1	......	......	......	......	......	......	......	......	......
Pepper	16	29	14	28	......	......	......	......	31	29
Cayenne	......	......	......	......	......	......	......	......	13	11
Total	53	73	34	32	48	86	38	112	103	118

* Many labeled mixtures.

It is seen that several years after the enactment of the law adulteration of spices is as enormous as at first. This, however, seems to arise largely from lack of prosecution and non-enforcement of the law.' Of the occurrence of adulterants in spices, the chief analyst says, in his report for 1885:

> During the year considerable attention was paid to spices and condiments. Viewing the fact that in the past a very large amount of adulteration had been reported as prevailing in these substances, and with a view to ascertaining whether the adulteration was practiced by the manufacturers or by the dealers, a systematic visitation was made of all the spice grinders in the Dominion (or of all that could be recognized as such), and their factories and stores were inspected under sections 7 and 8 with the results as shown in the appendix.
>
> The examination of nineteen samples of ground cinnamon resulted in finding seven genuine; four to consist of a substitution of cassia; one was adulterated with cassia, and six with other inert matter; one consisted of cassia adulterated with foreign vegetable matter.
>
> Of ground cloves, twenty-two samples were examined. Twelve proved to be pure and ten adulterated, the adulterant chiefly used being clove stems, pea-meal, roasted and ground cocoanut shells.
>
> Of sixty-six samples of ground ginger, twenty-nine were reported as being adulterated, almost exclusively with wheat flour, non-injurious to health, doubtless; but unless the purchaser be duly warned of the nature of the compound his pocket would be seriously prejudiced, if not injured, as this sophistication was practiced to the extent of from 10 to 15 to from 25 to 40 per cent., the pungency being imparted by the judicious admixture of cayenne pepper.
>
> Fifty samples of mustard were examined, and many of these were properly sold as "compound" or mixtures, but one of the worst samples was sold with a label guaranteeing it to be "ground from finest English seed, and free from adulteration." Of the fifty samples, nine were reported genuine, two of excellent quality, and thirty-nine were all, more or less, admixtures of mustard seed or mustard cake (from which the natural fixed oil had been expressed) with wheat flour and turmeric, and, in some cases, with corn-starch or bean meal, in varying proportions, up to as high as 50 or even 60 per cent. It was formerly contended that the addition of wheat flour or other inert matter was a necessity, to give the ground mustard keeping qualities and make the condiment palatable by softening its natural acidity. But the most reputable manufacturers have demonstrated the fallacy of this contention by the produc-

tion of an absolutely pure mustard, which has received public acceptance and appreciation, and two, at least, of our home manufacturers are happily following in their steps. It is a question yet to be decided how far the use of mustard cake, deprived of the natural fixed oil, is permissible in the manufacture of this condiment. Dr. Ellis' observations on this matter are very much to the point, and have received confirmation by similar experiments in my laboratory, and doubtless when next the analysts meet in conference this question will be settled in a manner favorable to the use of mustard cake.

Twenty-four samples of cayenne pepper were examined, of which fourteen were reported adulterated, but three of these were appealed to the judgment of the chief analyst, and the decision of the public analysts was not sustained, as will be seen on reference to "appeal cases." The remaining ten were reported adulterated with wheat flour and colored earth, in one case to the extent of 50 per cent. The other ten samples were reported unadulterated, save one, which was doubtful, it apparently having been artificially dressed with a fixed oil.

Sixty samples of ground pepper, black and white, were examined, of which thirty-one are reported as unadulterated, one doubtful, and twenty-eight all more or less adulterated—the generality of them to the extent of from 10 per cent. to 20 per cent., but the more flagrant cases from 30 up to even 75 per cent. in one case. The adulterant is chiefly farinaceous matter, also mustard husk, pepper hulls, clay, sand, and, not the least conspicuous, *ground cocoanut shells*—doubtless an innocent admixture, so far as health is concerned, but decidedly not a material of a character to improve the flavor or value of the pepper as a condiment.

As stated, these samples of spices were all obtained from either the actual producer or wholesale distributers; and the results prove that whether or not the retail vender still further "improves" his spices, &c., before retailing them, his demand for a cheap, adulterated article is amply provided for by the manufacturing dealer.

For the most part, the producers of these sophisticated goods expressed themselves anxious for the enforcement of the law, that, if sold, they should be distinctly labeled as impure. Some, on the other hand, contended that the public was benefited by a slight admixture—that a really better article could be supplied at a lower price if the finest and freshest spices were ground with an admixture of inert matter, than a thoroughly pure article, but ground from old or perished spices—a specious contention, utterly untenable in the true interests of the public.

But have not the producers of these sophistications some justification; is not the supply of a demand which, undoubtedly, has existed, a justifiable enterprise, whatever that demand may be, so long as it is within the law? Ignorance does, undoubtedly, demand cheapness, and a demand thus ignorantly made is only too surely supplied, and hence the need for costly legislation to protect an ignorant and thoughtless public against itself, for it does demand the very goods which the analyst must condemn and the vender be prosecuted and fined for selling, whereas the public's reckless ignorance is the chief cause, and should suffer some measure of the penalty. It is time that through the operation of this act such ignorance should be cleared away, and the public be enlightened and awakened to its own true interests.

These remarks apply equally well to much of the spices sold in the United States. Massachusetts, New York, New Jersey, and Michigan alone have laws* of any value in regard to the adulteration of food, and it is easy to see in what condition the spices and condiments sold in other parts of the country must be. In Massachusetts, where investigations under the law have been going on since 1882, it has been shown, as in other localities, that the adulterants of spices are numerous but harmless. Dr. Sharpless, in his report of 1882 upon the subject, remarks,

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* See Appendix.

that he agrees with the opinion expressed by Dr. Leeds, of New Jersey, in his report of 1880 to the New Jersey State board of health, that there has been much sensational writing upon the subject. This is perhaps the case with some few writers of that stamp, but it can have done no harm, for it has not produced sufficient effect upon the public to create a demand for any purer spices, as appears from the figures of Dr. E. S. Wood in 1882, who reports in regard to the samples he examined as analyst of foods for Massachusetts:

Articles.	Genuine.	Adulterated.	Per cent. adulterated.
Black pepper	20	44	68.75
White pepper	13	31	70.45
Red pepper	5	...	...
Mustard	29	47	61.84
Ground cloves	0	11	100.00
Cassia	0	2	100.00
Pimento	2	...	...
Ginger	1	...	...

This is nearly as serious a condition as was found in Canada, but again, in 1885, Dr. Wood reports:

Articles.	Genuine.	Adulterated.	Compounds.	Per cent. adulterated.
Mustard	31	37	20	64.8
Cloves	15	76	...	83.5
Cassia and cinnamon	48	26	1	36.0
Ginger	55	17	...	23.6
Allspice	30	8	...	21.1
Ground mace	9	9	...	50.0
Black pepper	6	19	3	78.6
White peper	21	39	...	65.0

The above is little or no improvement. He found the common adulterants of mustard to be flour, turmeric, and sometimes a little cayenne. Cloves suffered from extraction of the volatile oil and the addition of clove stems, allspice, burnt shells, and other cheap substances. Cassia contained ground shells and crackers; ginger was in many cases colored, and in some instances wheat and corn flour and clove stems were present. Allspice is too cheap to be often adulterated, but in eight samples mustard hulls, ground shells, clove stems, and cracker dust were found. In mace, flour and cornmeal were diluents, and for the peppers, crackers, mustard hulls, pepper dirt, powdered charcoal, rice, corn, and buckwheat.

Under the New York law of 1881, Prof. S. A. Lattimore investigated a number of spices and spice mixtures submitted to him.

Professor Lattimore's report furnishes abundant proof in support of the common impression regarding the adulteration of spices.

On the results of his examination of the commercial ground spices he comments, after giving the proportions which were found adulterated, in these words:

Articles.	Samples examined.	Adulterated.	Percentage of adulteration.
Mustard	18	12	66.6
Ginger	15	9	00.0
Allspice	27	19	70.4
Cinnamon	22	18	81.8
Cassia	7	4	57.1
Cloves	21	16	76.2
Pepper:			
Black	40	28	70.0
White	7	5	71.4
Red	10	5	50.0
Mace	8	4	50.0
Nutmeg	5	2	40.0

As the above table shows, a large proportion of them are adulterated, and that with substances presenting a certain uniformity. The spices present an inviting field for the exercise of fraudulent arts. They are almost universally sold in the form of fine powder and in opaque packages, which do not admit of easy examination on the part of the purchaser. Consequently any cheap substance which may be easily pulverized to a similar degree of fineness, and which possesses little distinctive taste or odor of its own, answers the purpose; so that the list of adulterants for this class of articles is naturally very large. The adulterations found in the samples now under consideration may be classed into four groups. First, integuments of grains of seeds, such as bran of wheat and buckwheat, hulls of mustard seed, flax seed, &c. Second, farinaceous substances of low price, such as are damaged by the accidents of transportation or long storage—such as middlings of various kinds, corn-meal, and stale ship's bread. Third, leguminous seeds, as peas and beans, which contribute largely to the profit of the spice mixer. Fourth, various articles chosen with reference to their suitableness for bringing up the mixture as nearly as possible to the required standard of color of the genuine article. Various shades, from light colors to dark browns, may be obtained by the skillful roasting of farinaceous and leguminous substances. A little turmeric goes a great way in imparting the rich yellow hue of real mustard to a pale counterfeit of wheat flour and terra alba, or the defective paleness of artificial black pepper is brought up to the desired tone by the judicious sifting in of a little finely pulverized charcoal. Enough has been already given to show that the field for sophistications of this sort is a wide one, and offers large scope for the development of inventive genius; so that each manufacturer of articles of this class would be likely to possess his own trade secrets. It will be observed that the adulterating materials just mentioned all belong to the class claimed to be harmless. In no instance has any poisonous substance been discovered. The proportion of foreign and genuine substances in the spices varies between wide limits, in some instances the former being slight; in others, the latter seemingly present in just sufficient quantity to impart faintly the requisite taste or odor. Even this small proportion of the professed article is occasionally further diminished by the substitution of other substances; as, for example, in imparting to corn-meal finely ground a pungency suggested by real ginger by the addition of a little salt and red pepper.

It is probably not so widely known as it should be that the demand for the materials for adulteration has called into existence a branch of manufacturing industry of no insignificant magnitude, having for its sole object the production of articles known as "spice mixtures" or "pepper dust." The use of "pepper dust," or, as the article is commonly designated in the technical language of the trade by its abbreviation "P. D.," is a venerable fraud.

> The manufacture of "P. D." is now a regular branch of business, and the original and specific term "pepper dust" has expanded with the progress of inventive art to generic proportions, until now we have as well-known articles sold by the barrel "P. D. pepper," "P. D. ginger," "P. D. cloves," and so on through the whole aromatic list. When it is considered that these imitations, lacking only such flavoring with the genuine article as the dealer thinks necessary to make his goods sell, are sold at from three to four cents a pound, and the retail price paid by the consumer is compared with it, the strength of the temptation to engage in such practices is clearly seen. When manufacturers openly advertise themselves as assorters and renovators of merchandise, and openly propose to cleanse musty and damaged beans by a new and patented process, it is full time that its significance should be considered by the public.

From the investigations, which have been quoted, it appears that the adulterants which are met with in this country are very numerous. Under the head of spice mixtures, or "P. D.," much refuse of all descriptions is used up, and there are such changes in the character of the material from time to time as the sources of damaged material or refuse at hand may suggest. The diluents used in Baltimore and in the District of Columbia seem to be different from those in New York, and, in consequence, some of the adulterants which are mentioned most commonly in the reports from the North are not found in the spices of southern millers. While it is possible, therefore, to give a list of substances which have been used as adulterants, it is quite out of the question to say in what directions the ingenuity of spice-mixers will extend in the future. The following contaminations in the various spices have been already noted in this country:

Spices.	Adulterants.
Allspice	Spent cloves, clove stems, cracker dust, *ground shells* or charcoal, *mineral color yellow corn.*
Cayenne	*Rice flour*, salt and ship-stuff, *yellow corn*, *turmeric*, and *mineral red.*
Cassia	*Ground shells* and *crackers*, *turmeric*, *minerals.*
Cinnamon	*Cassia*, peas, starch, *mustard hulls*, *turmeric*, *minerals*, *cracker dust*, *burnt shells* or charcoal.
Cloves	*Spent cloves, clove stems*, *minerals*, allspice, roasted shells, wheat flour, peas.
Ginger	*Cereals*, *turmeric*, *mustard hulls*, *cayenne*, peas.
Mace	Cereals or starch, buckwheat, *wild mace.*
Nutmeg	Cereals or starch, wild nutmeg.
Pepper	*Refuse of all sorts*, *pepper dust*, *ground crackers*, *or ship stuff*; *rice*, *mustard hulls*, *charcoal*, cocoanut shells, *cayenne*, beans, *bran*, *yellow corn.*
Mustard	*Cereals and starch*, *turmeric*, peas, *yellow corn meal*, *ginger*, *gypsum.*

The materials in italics have been identified in spices examined in the laboratory of this division, but some of the commoner adulterants have not been found. Considering the spices individually, there are certain peculiarities, as they are met with pure and in the trade, which should not be overlooked.

MUSTARD.

Mustard, as sold in the ground state, should be the farina or flour of the black or white mustard seed—that is to say, the flour from the interior of the seed bolted or separated from the hulls. The two kinds of seed, although derived from plants of the same genus, are somewhat different in their chemical composition. The black seed is much the most pungent, and develops, on mixing with water, a volatile oil, which gives this condiment its penetrating character. There is also present in the seed a complicated organic substance of a bitter nature, to which is due some of the peculiar flavor, and while the white seed forms no volatile oil with water, it contains more of the bitter substance. It is, therefore, very common to mix the two in grinding. The sources of the seed are various. In our markets at present there are quoted California black and white, Dutch, Trieste black, and English—the last being the most valuable.

In the manufacture of the seed into flour for the market, two customs have arisen which change the nature of the original substance, and therefore would commonly come under the head of adulteration. One is extremely old, the addition of wheat flour for the purpose of making the condiment keep better. This necessitates the restoration of the yellow color by turmeric, or other dye-stuff. These diluents are harmless as a rule, but there seems to be no reasons for their use, and it is gradually becoming commoner to find mustard free from them in English brands.

The other custom is the abstraction of the fixed oil by pressure before grinding the seed. The percentage of this oil is over 30. It adds nothing to the flavor of the mustard, probably injures its keeping qualities, makes the seed more difficult to mill, and its removal is therefore a benefit. It is a nearly universal custom at the present day in this country, and is not considered as fraudulent by the Canadian analysts.

Falsifications of mustard other than those mentioned are not common, although gypsum has been found in low-grade mustard and several other adulterants, among them ginger of low grade. The hulls bolted from the flour in the process of manufacture are preserved and form the basis of the adulteration of many other spices.

PEPPER, BLACK AND WHITE.

Pepper is more in demand than any other spice, and in consequence is more adulterated. Its appearance in the ground form, especially of the black, is such as to make it possible to use all sorts of refuse for this purpose, and almost everything that has been used as an adulterant has been found in pepper. White pepper, which is simply the black deprived of its outer black coats, is, of course, less easily falsified; but in France is diluted to an immense extent with ground olive stones, which bear a striking resemblance. Among the samples from Washington grocers, pepper sweepings—that is, husks and dirt—rice or corn, and mustard-hulls were the commonest admixtures. Sand is said to be very commonly added abroad, but has not been met with here. In Canada and New York ground cocoanut-shells are a cheap source of adulteration, but they have not extended so far south.

Specimens from Baltimore mills of very low-quality goods were found to contain but little pepper, and that of the worst quality, being made up of cracker dust, yellow corn, cayenne, and charcoal in so disgraceful a way as to be visible to the unassisted eye on close examination.

The quality of a ground pepper can be told by an expert from its weight and color, and on examination with a lens of low magnifying power. The particles are not coarsely ground, and it is not difficult to pick out pieces of husk, yellow corn, and rice, and, if necessary, a more careful investigation under a microscope of higher power will serve for confirmation. Black pepper, in our experience, is much more liable to adulteration than white, although it is perfectly easy to dilute the latter with broken rice or cracker dust, which are inexpensive. All these materials, fortunately, owing to the grossness of the adulteration, are readily recognized, and there is hardly the necessity for recourse to chemical analysis. There has been, however, considerable investigation in this direction, so that there are means of confirming the optical examination which are of great value. Determination of the amount of starch is one of the methods upon which some reliance can be placed; for, if under the microscope foreign starch is not detected, then the addition of "P. D." or other starch free adulterants, will diminish the percentage found, and the reverse. In this way, too, one is able to arrive at an approximate conclusion as to the proportion of adulterants added, which can only be estimated within wide limits under the microscope.

In spite of the immense amount of adulteration, it is possible, from the best shops, to obtain pure ground peppers, but it is at the same time safer with a family spice-mill to grind the whole berries as they are needed. The sources of our pepper supply are Tellicherry on the west coast of Hindostan, which is graded high, and Penang and Singapore for the east, Sumatra, Java, &c. The importations are principally through London, and not direct. The supply of ground pepper from England will usually be found more pure than our own brands, and at the same time is naturally more expensive.

CAYENNE OR RED PEPPER.

This condiment should consist of the ground pods of any of several species of *Capsicum*, known as chilies or peppers. It is said to have been adulterated with many substances, brick dust, red lead, and coloring matters; but this has only been found to be the case in two cheap Baltimore cayennes, while in Washington only rice has been detected, but that quite frequently. Inferior material is no doubt often ground, but the small value of the pods and the small quantity consumed does not tend to increase adulteration.

GINGER.

Ginger is the root, or, technically, rhizome, of a plant somewhat similar in appearance to our iris and flag. It is grown in various parts of the world and prepared with great care and great carelessness, being at times scraped and bleached, at others simply dried in any condition, so that there is an immense number of varieties and qualities to be found in the market. They all, however, retain sufficiently the marked peculiarities of the starchy fibrous root to make the detection of adulterants easy. The common ones are the addition of wheat flour or some starch as a diluent, the coloring with turmeric to suit a popular fancy for gingerbread or of spent material which has been used in making tincture. Mustard hulls and cayenne are also found in some cases, but have seldom been detected here. They are added to give pungency and make up for the addition of flour. Their detection is easy. The sources of our supply are Jamaica and the West Indies, Cochin China, Africa and India. Jamaica is the best and most carefully prepared.

CLOVES.

The flower buds of the clove tree carefully picked and dried constitute the spice known by that name. Their valuable properties are due to the volatile oil which they contain, the best having as much as 16 per cent.

The removal of this oil is so very easy that it is the commonest method of deception to do so before grinding the spice and to then dispose of it as pure. We have ready means of determining the loss chemically, but the microscope gives no indications. The addition of the cheaper clove stems is also practiced, as they cost but 6 cents when the buds cost 27. The microscope reveals their presence by certain cells which they contain which are absent in the buds.

Pimento is sometimes substituted in part or entirely, as it has a clove-like flavor but only 4 or 5 per cent. of volatile oil. It is worth less than one-fifth the price of cloves. Its chemical composition and its structure, that of a berry, reveals its presence. The addition of the coarser adulterants, mineral matter, cocoanut shells, flour, peas, and the like, have only been observed in two instances, but no doubt frequently occur, as has been found in Canada.

The sources of our supply are the East Indies (Amboyna), African (Zanzibar), and American, ranking in value in the order named. Cloves should, if possible, be always purchased whole, as they deteriorate less readily in that form.

CINNAMON AND CASSIA.

These spices are the barks of several species of the genus *Cinnamomum*, the true cinnamon being a native of Ceylon, where it is largely cultivated, and the cassias being derived from several other species growing in China, India, and the East Indies. Cinnamon as it reaches the market is very thin, the outer and inner coats of the bark having been removed. Cassia on the other hand is thick, as it consists of the entire bark and can be distinguished by its retaining its natural outer surface. Cinnamon is by far more valuable than the cassia, as there is a smaller supply and intrinsically it contains a much greater proportion of volatile oil and that of higher and more delicate aroma. In consequence cassia is largely substituted for cinnamon, and in fact not a particle of ground cinnamon can be found in the market. It can be found in the whole condition in good quality only in drug stores. Cassia exists in many forms and qualities, and sells at wholesale at from 7 to 40 cents a pound. That known as Saigon is the best, and that exported from Batavia the poorest. Cassia buds also hold a small place in the market.

The detection of the substitution of cassia for cinnamon, since the barks are of trees of the same species, is more difficult than is usually the case and may prove troublesome to a novice. The presence of more woody fiber in the latter with the aid of chemical analysis serves, however, as a reliable distinction. In the samples which have come into our hands not a particle of material labeled ground cinnamon proved to be anything other than cassia. The spice millers appeared, however, to be satisfied to stop at this point and in only one case was there addition of cheap stuff to the cassia. When added there is no difficulty in detecting it as has been done here and in Canada, where peas, starch, ground shells, and crackers have been found in powder labeled both cassia and cinnamon.

The barks can, in most cases, and especially the cinnamon, be used nearly as well in the whole condition and should at least be so purchased and then ground. A slight acquaintance with the appearance of the different qualities will teach one the proper selection to make.

NUTMEG AND MACE.

These spices are different portions of the fruit of a tree known as the nutmeg tree, *Myristica fragrans*, the nutmeg being the inner kernel and the mace one of the outer coats or arillus. The tree grows principally on the Banda Islands and the spices reach us through London. They can always be obtained in their original condition and should be so purchased. When ground they are mixed with diluents of various descriptions, principally cereals and their refuse, which are easily detected.

Owing to the infrequency of the sale of the powdered nutmeg and mace, their adulteration has attracted but little attention.

SOURCES OF OUR SPICE SUPPLY.

Although the countries where the spices are grown have been already given in a general way and in a later chapter will be given more in detail, these countries, in many instances, are not the direct sources of our supplies. For instance, of the pepper imported in the fiscal year ending June 30, 1886, amounting to nearly 12,000,000 pounds, over 8,000,000 came to us from England, and about 3,000,000 from the British East Indies, including the ports of Singapore and Penang. In regard to the other spices data are found in the annual statement of the Bureau of Statistics of the Treasury Department and in some additional tables furnished to this office by the Bureau, which are here given:

Imports of unground spices, free of duty, for the year ending June 30, 1886.

Countries from which imported.	Nutmegs.		Pepper.		All other.	
	Quantity.	Value.	Quantity.	Value.	Quantity.	Value.
	Pounds.		*Pounds.*		*Pounds.*	
Argentine Republic						
Austria			2,128	$225		
Belgium						
Brazil					194	$32
Central American States:						
Costa Rica						
Guatemala			12	1		
Honduras						
Nicaragua						
San Salvador						
Chili			5,905	498		
China	11,825	$3,982	344,552	45,808	257,402	21,737
Denmark						
Danish West Indies						
Greenland, Iceland, and the Faroe Islands						
France						
French West Indies	300	120				
French Guiana						
Miquelon, Langley, and Saint Pierre Islands						
French Possessions in Africa and adjacent islands						
French Possessions, all other						
Germany	4,740	2,293			16,831	1,832
England	247,839	98,412	8,214,037	1,157,264	2,005,255	176,535
Scotland						
Ireland						
Gibraltar						
Nova Scotia, New Brunswick, and Prince Edward Island						
Quebec, Ontario, Manitoba, and the Northwest Territory			1,465	338	740	35
British Columbia						
Newfoundland and Labrador						
British West Indies	3,344	889	480	9	3,017,943	146,880
British Guiana						
British Honduras						
British East Indies	464,862	179,169	2,927,472	403,090	1,006,938	66,536
Kong-Kong					1,089,089	91,650
British Possessions in Africa and adjacent islands			11,984	1,042	1,563,073	59,101
British Possessions in Australasia						
British Possessions, all other			840	66		
Greece						
Hawaiian Islands						
Hayti						
Italy						
Japan						
Liberia					4,233	296
Mexico	50	37	48,255	7,528	907	100
Netherlands	210,635	83,412			31,038	7,292
Dutch West Indies						
Dutch Guiana						
Dutch East Indies	245,912	90,065	3,261	360	817,756	35,618
Peru						
Portugal						
Azore, Madeira, and Cape Verde Islands						
Portuguese Possessions in Africa and adjacent islands						
Roumania						
Spain					151	36
Cuba					856	241
Porto Rico					101,338	4,302
Sweden and Norway						
Switzerland						
Turkey in Europe						
Turkey in Asia						
Venezuela					1,160	89
All other countries and ports in Asia						
All other countries and ports in Africa			283,062	28,154	851,307	66,624
Total	1,189,507	458,379	11,843,453	1,644,383	10,767,211	678,936

Missing Page

Statement showing, by customs districts and ports, the quantities and values of spices imported and entered for consumption, &c.—Continued.

FREE OF DUTY.

Customs districts and ports.	Cloves.		Clove stem.		Ginger root.	
	Quantity.	Value.	Quantity.	Value.	Quantity.	Value.
	Pounds.		*Pounds.*		*Pounds.*	
Aroostook, Me	....	....	....	....	....	....
Baltimore, Md	11,098	$1,500.00	....	....	88,214	$4,961.00
Boston, Mass	20,708	2,632.00	30,000	$675.00	954,716	38,664.00
Brazos de Santiago, Tex	....	....	....	....	....	....
Chicago, Ill	6,606	1,011.00	....	....	95	1.00
Cincinnati, Ohio	3,931	601.00	....	....	....	....
Corpus Christi, Tex	....	....	....	....	....	....
Detroit, Mich	....	....	....	....	....	....
Duluth, Minn	742	35.00	....	....	....	....
Galveston, Tex	....	....	....	....	....	....
Key West, Fla	30	6.20	....	....	....	....
Middletown, Conn	....	....	....	....	....	....
Minnesota, Minn	....	....	....	....	....	....
New Orleans, La	....	....	....	....	....	....
New York, N. Y	1,234,576	145,634.00	266,127	5,057.00	3,192,245	174,178.00
Oregon, Oreg	....	....	....	....	420	4.00
Oswegatchie, N. Y	....	....	....	....	....	....
Paso del Norte, Tex	....	....	....	....	....	....
Philadelphia, Pa	13,091	1,732.00	....	....	34,534	2,392.00
Saluria, Tex	....	....	....	....	....	....
San Francisco, Cal	....	....	....	....	....	....
Saint Louis, Mo	8,101	1,232.00	....	....	986	51.00
Willamette, Oreg	....	....	....	....	5,900	194.00
Total	1,298,883	154,383.20	296,127	5,732.00	4,277,110	220,415.00

FREE OF DUTY.

Customs districts and ports.	Mace.		Nutmegs.		Pepper, white and black.	
	Quantity.	Value.	Quantity.	Value.	Quantity.	Value.
	Pounds.		*Pounds.*		*Pounds.*	
Aroostook, Me	....	....	....	....	....	....
Baltimore, Md	....	....	22,314	$8,634	429,849	$57,075
Boston, Mass	6,733	$1,907.00	66,465	27,462	476,990	54,951
Brazos de Santiago, Tex	....	....	....	....	460	102
Chicago, Ill	....	....	....	....	23,746	3,575
Cincinnati, Ohio	....	....	....	....	139,726	20,424
Corpus Christi, Tex	....	....	....	....	....	....
Detroit, Mich	....	....	....	....	....	....
Duluth, Minn	....	....	....	....	....	....
Galveston, Tex	....	....	....	....	....	....
Key West, Fla	115	11.50	....	....	....	....
Middletown, Conn	....	....	....	....	....	....
Minnesota, Minn	....	....	....	....	....	....
New Orleans, La	....	....	....	....	....	....
New York, N. Y	115,022	28,617.00	1,008,282	395,583	9,119,301	1,309,084
Oregon, Oreg	....	....	....	....	....	....
Oswegatchie, N. Y	....	....	....	....	1,465	338
Paso del Norte, Tex	....	....	....	....	....	....
Philadelphia, Pa	139	118.00	177	162	195,455	25,702
Saluria, Tex	....	....	....	....	25	8
San Francisco, Cal	....	....	86,014	23,643	509,882	71,296
Saint Louis, Mo	....	....	6,204	2,858	98,887	15,255
Willamette, Oreg	....	....	....	....	....	....
Total	122,009	30,653.50	1,189,456	458,342	10,995,786	1,557,810

Statement showing, by customs districts and ports, the quantities and values of spices imported and entered for consumption, &c.—Continued.

FREE OF DUTY.

Customs districts and ports.	Pepper, cayenne.		Pimento.		All other, n. s. e., or p. f.	
	Quantity.	Value.	Quantity.	Value.	Quantity.	Value.
	Pounds.		*Pounds.*		*Pounds.*	
Aroostook, Me						
Baltimore, Md						
Boston, Mass			130	$4	11,585	920.00
Brazos de Santiago, Tex						
Chicago, Ill						
Cincinnati, Ohio						
Corpus Christi, Tex	9,552	$1,296			37,446	6,111.00
Detroit, Mich						
Duluth, Minn						
Galveston, Tex						
Key West, Fla					54	5.20
Middletown, Conn						2,400.00
Minnesota, Minn						
New Orleans, La						
New York, N. Y	723,459	73,789	2,500,523	109,605	33,600	
Oregon, Oreg						
Oswegatchie, N. Y						
Paso del Norte, Tex	1,081	47				
Philadelphia, Pa	79,118	5,503	130	6		
Saluria, Tex						
San Francisco, Cal					151,313	21,511.00
Saint Louis, Mo					16,329	1,265.00
Willamette, Oreg						
Total	813,210	80,635	2,500,783	109,615	250,327	32,212.20

DUTIABLE.

Customs districts and ports.	Mustard, ground or preserved, in bottles, or o.		All other, ground or produced, n. s. e. or p. f.	
	Quantity.	Value.	Quantity.	Value.
	Pounds.		*Pounds.*	
Aroostook, Me	4½	$1.35		
Baltimore, Md	10,660	2,839.00		
Boston, Mass	24,670	7,074.00	894	$392.00
Brazos de Santiago, Tex				
Chicago, Ill				
Cincinnati, Ohio				
Corpus Christi, Tex			6	1.00
Detroit, Mich	896	278.00		
Duluth, Minn	5	1.00	20	5.00
Galveston, Tex	1,800	317.00		349.41
Key West, Fla	3	.42	2,762	246.00
Middletown, Conn			3,312	9.00
Minnesota, Minn			90	3,704.00
New Orleans, La	45,207	8,810.00	36,982	498.00
New York, N. Y	404,099	100,609.00	2,295	
Oregon, Oreg				
Oswegatchie, N. Y				
Paso del Norte, Tex			442	70.62
Philadelphia, Pa	106,735	30,701.00	420	157.00
Saluria, Tex				
San Francisco, Cal	38,985	11,195.00		
Saint Louis, Mo	4,490	1,111.00		
Willamette, Oreg			94½	5.26
Total	637,555½	162,936.77	47,317½	5,431.29

Unfortunately, with the exception of pepper and nutmeg, the tables giving the countries from which the imports were made do not distinguish between the spices. We are, however, able to see that a large portion of our supply comes through England, and the effect of this upon its quality is certainly not to improve it. The amount of each spice entered for consumption is visible in the last table and the port of arrival as well. New York of course receives the largest portion, followed by Boston and Baltimore, which are both milling centers.

As the spices are offered by the wholesale merchant they have a variable value, the quotations in New York for the week ending December 27, 1886, being as follows:

Kind of spices.	Price per pound.	Kind of spices.	Price per pound.
	Cents.		*Cents.*
Cassia:		Nutmegs, 110s	48 to 50
Batavia	7 to 7½	Pepper:	
China	5¼	Singapore	18
Saigon:		West coast	15½ 16
Rolls	30 40	Acheen, prime	15
Broken	30	Penang, white	25 26
Chips	9 10	Singapore, white	28½ 29
Buds	10¼ 10½	Red:	
Cloves:		Zanzibar	8½ 9
Prime	27	Bombay	6
Amboyna	27	Pimento, prime	5
Stems	6	Mustard:	
Ginger:		California:	
African	5	Brown	3¾ 4¼
Calcutta	3¾ 4	Yellow	3¼ 4½
Cochin	10 13	Dutch	5¾ 6
Mace:		English	6¼ 6½
Batavia	50	Trieste, brown	5½ 5¾
Banda	50		
Penang	50		

It is of interest to examine these figures and compare them with the prices of wholesalers and retailers of ground spices.

The following figures show that prices alone are often a good indication of adulteration, the ground article being sold at wholesale or even retail for less than the cost of the pure unground spice.

Pure whole spices	Price per pound.	
	Wholesale.	Small lots.
Ginger root:	*Cents.*	*Cents.*
Bleached:		
Jamaica	....	16
American	....	14
Unbleached:		
Jamaica	....	14
Cochin	7½ to 11	12
Calcutta	3¾ 4	8
Clove stems	6	10
Clove:		
Singapore	26½ 27	30
Amboyna	27	32
Pepper:		
White Singapore	24½	32
Best west coast	10½ 16	18
Acheen	15¾	18
Mustard seed:		
Brown Trieste	5½ 5¾	12
Yellow English	6¼ 6½	12
California	3¼ 4½	8
Brown California	3¾ 4¼	8
Cassia bark:		
Batavia	7 7½	10
Saigon stick	36 40	42
Saigon chip	9 10	10
Ligna sticks	....	9
Singapore black pepper	17½ 17¾	....
Zanzibar red pepper	9½	10

Prices as supplied by spice mills.

Name.	Price per pound.	
	Wholesale, barrels.	Small lots in tin.
	Cents.	*Cents.*
Durham mustard	12	16
Extra strong mustard	16	20
Extra American mustard	18	22
B. Pure Pepper	12	16
Best pepper	10	14
Pure cinnamon	12	16
Best cinnamon	10	14
Pure ginger	7	11
Best ginger	6	10
Pure cayenne	12	16
Best cayenne	16	14
Pure allspice	6	10
Best allspice	5	9
Pure cloves	20	24
Best cloves	17	21
Pure mace	50	54
Pure white pepper	25	29

CHARACTER OF THE SPICES IN THE DISTRICT OF COLUMBIA.

The spices found in Washington are from various markets. The first class grocers carry the best English and rarely good American brands. Adulteration, however, is frequent, especially among the mustards, peppers, and cinnamon, the first having lost its oil and added flour, and the last having cassia substituted for it. Among the cheaper class of dealers adulterated spices are nearly universal, the supply being obtained largely from Baltimore and to a small extent ground in Washington, both places in which yellow corn and charcoal are much used as adulterants.

Of a series of samples collected impartially from all classes of shops the ratio of adulterated to non-adulterated was as follows:

Variety of spice.	Pure.	Adulterated.	Substituted.	Inferior or suspicious.
Cassia	3			1
Cinnamon			10	
Cloves		2		9
Ginger	4	4		
Mace	3	2		
Mustard		10*		1
Nutmeg	3			
Pepper:				
Black	1	9		
White	2	2		
Red	1	5		
Pimento	5	3		1

*Oil expressed in one case and tumeric added and oil expressed in all American brands.

The preceding samples, which have proved to be so largely adulterated, have been used in connection with a collection of authenticated whole spices obtained directly from the importers as a means of investigating the methods which have been recommended for the detection of adulterants, and the results of our examination of published methods and our own work are presented in the following pages, giving to the analyst a large amount of technical and scientific information which is of less interest to the general reader.

PART II.

THE DETECTION OF ADULTERATION OF SPICES AND CONDIMENTS.

In attempting to detect the adulteration of spices and condiments the methods which can be employed are of three kinds and depend upon the differences in structure between the adulterants and the substances to which they are added, and upon their proximate composition. The former differences are recognized by mechanical separation and the use of the microscope and the latter by chemical analysis. In the use of the microscope a knowledge of and ability to recognize the principal tissues which constitute the particular plant parts which are used as spices and also of those used as adulterants is necessary, while in the chemical examination the principles of proximate analysis must be understood and applied.

It is as necessary that the analyst should be thoroughly acquainted with the application of the microscope to the determination of cellular structure as to be able to make determinations of proximate principles in the substances under examination. In fact, a mechanical separation and microscopical examination is much more expeditious and more at the command of the majority of persons searching for adulteration.

Chemical analysis requires a systematic and extensive investigation of large numbers of samples, both pure and adulterated, to fix a standard of comparison, and this has hitherto been seldom done, owing to the elaborate nature of the work and the expense involved. It should not be neglected, however, since it serves as a most certain confirmation of the microscopic results, besides furnishing information in regard to the quality of the specimen examined and as to the quantity of any adulterant, which cannot be obtained in any other way. While, therefore, the microscopic method will always retain its value for preliminary and qualitative examinations, it must, with the development of the means of chemical investigation, become more and more a mere adjunct of the latter, as in fact the microscope has become in all branches of science. The application of the microscope to the detection of adulterants will, therefore, be considered first.

MECHANICAL SEPARATION AND MICROSCOPIC EXAMINATION OF SPICES AND CONDIMENTS.

As a preliminary to the microscopic examination a mechanical separation by means of sieves of different mesh furnishes a means of detecting adulterants and selecting particles for further investigation, which is of the greatest value and often reveals without additional means the presence of foreign materials. Many adulterants are not ground as fine as the spice to which they are added, and by passing the substance through a sieve of from 40 to 60 meshes to the inch the coarser particles remaining will either be recognized at once by the unaided eye or with a pocket lens or the microscope. In this way turmeric is readily separated from mustard, and yellow corn, mustard hulls and cayenne from low-grade peppers; in no case was the aid of more than an ordinary pocket lens necessary for subsequent recognition, although higher powers of the microscope were confirmatory.

Without entering here into the practical manipulation of the microscope, it may be said that for the purposes of the food analyst it is only necessary to have a stand of good workmanship, not necessarily, though preferably, furnished with substage condenser, but supplied with Nicol prisms for the use of polarized light. Objectives of inch, half-inch, or, for some of the starches, one-fifth inch equivalent focus are sufficient. One eye-piece of medium depth is also enough. It is also desirable to be provided with a dissecting microscope for selecting particles for examination from large masses of ground spice. For those who can afford it, the large stand of Zeiss made for this purpose proves most useful, but simpler forms or even a hand lens will serve perfectly well.

For smaller apparatus it is unnecessary to provide anything aside from what is found in ordinary laboratories. A few beakers, watch glasses, stirring rods, and specimen tubes, with bottles for reagents will be sufficient in addition to the ordinary glass slides and cover glasses.

The reagents which are required include, in case no permanent mounts are required:

Alcohol, strong.
Ammonia.
Chloralhydrat, solution 8 parts to 5 of water.
Glycerine.
Iodine solution: water 15 parts, iodide of potash 20 parts, iodine 5 parts.
Water, distilled.
Schulze's reagent, a mixture of chlorate of potash and dilute nitric acid prepared as wanted.

Balsam in benzol and glycerine jelly are desirable for mounting media and some sheet wax for making cells.

In addition the analyst should supply himself with specimens of whole spices, starches, and known adulterants which can be used to become acquainted with the forms and appearances to be expected. It is easier to begin one's study in this way on sections prepared with the knife than upon the powdered substance, and it is often necessary to refer to them for comparison in the examination of trade samples.

PHYSIOLOGICAL STRUCTURE IN THE SPICES AND THEIR ADULTERANTS.

The vegetable tissues which made up the structure of the spices, and the material of a vegetable origin which are added as adulterants, consist of cells of different forms and thickness. Those which are most prominent and common are the parenchyma, the sclerenchyma, fibrous tissue, and the fibro-vascular bundles. Spiral and dotted vessels are also common in several of the adulterants, and in the epidermis other forms of tissue which it is necessary to be well acquainted with though not physiologically. The parenchyma is the most abundant tissue in all material of vegetable origin, making up the largest proportion of the main part of the plant. It is composed of thin-walled cells, which may be recognized in the potato and in the interior of the stems of maize. In the latter plant, also, the fibro-vascular system is well exemplified, running as scattered bundles between the nodes or joints, and easily made out.

Fibrous tissue consists of elongated thick-walled cells or fibers which are very common in the vegetable kingdom and are well illustrated in flax. They are not as common in spices as in the adulterants. They are optically active, and in the shorter forms somewhat resemble the cells next described. They are seen in one of the coats of buckwheat hulls and in the outer husk of the cocoanut.

The sclerenchyma is found in the shells of many nuts and in one or two of the spices. The cells are known as stone cells, from the great thickening of their walls, and to them is due the hardness of the shell of the cocoanut, the pits of the olive, &c. Their structure is illustrated in Fig. 5 from Strasburger.

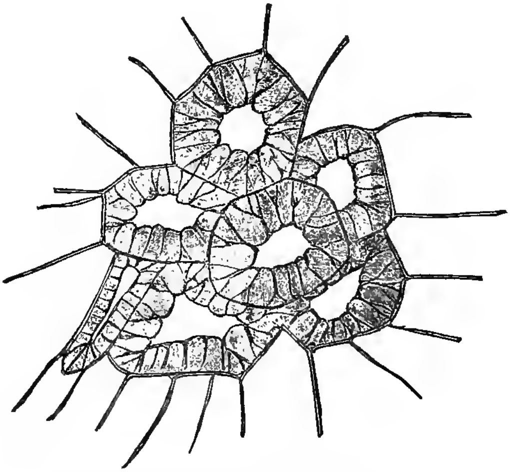

FIG. 5. Sclerenchyma or stone cells. × 240. (After Strasburger.)

Spiral and dotted vessels are common in woody tissue and are readily recognized. With all these forms the analyst should familiarize himself, and as an aid may consult Bessey's Botany in the American Science Series.

In pepper and in mustard the parenchyma-cells are prominent in the interior portion of the berry, while those constituting the outer coats are indistinct from their deep color in the pepper, but in the mustard characteristic of the particular species. In fact, in many of the spices, especially those which are seeds, the forms of the epidermal cells are very striking, and even if no attempt is made to classify them their peculiarities must be carefully noted, as the recognition of the presence of foreign husky matter depends upon a knowledge of the normal appearance in any spice. The fibro-vascular bundles are most prominent in ginger and in the barks, where in the powdered spices they are found as stringy particles.

The sclerenchyma or stone cells, are commoner in the adulterants, especially in cocoanut shells, where may also be seen numerous spiral cells and in the exterior coats fibrous tissue.

As aids to distinguishing these structures, the following peculiarities may be cited.

The stone cells and fibrous tissue are optically active and are therefore readily detected with polarized light, shining out in the dark field of the microscope as silver white or yellowish bodies.

The fibro-vascular bundles are stained deep orange brown with iodine, owing to the nitrogenous matter which they contain, while parenchyma is not affected by this reagent aside from the cell contents, nor has it any action on polarized light, remaining quite invisible in the field with crossed prisms.

STARCH.

Aside from the cellular tissue, starch is the most important element in the plant for the analyst, and its peculiarities will be considered quite fully.

It possesses an organized structure and is distinguished by its reaction with iodine solution, with which it strikes a deep blue or blackish blue color, varying somewhat with different kinds of starch and with the strength of the reagent.

Conversely its absence is marked by no blue color under the same circumstances. Heat, however, as in the process of baking, so alters starches, converting them into dextrine and related bodies, that they give a brown color with iodine instead of a blue black. They are then in fact no longer starch, although their form, often not being essentially changed, permits of their identification.

Although a practical experience in recognizing the starches by these characteristics is essential for their rapid detection when occurring as adulterants, a valuable guide may be supplied to a certain extent by artificial classifications, such as Vogel, Muter, and Blyth, after Tripe's work, have arranged.

Vogel's and Muter's are based on the form and size of the granules, of the hilum or central depression or nucleus and the prominence and position of the rings. Tripe showed that with polarized light and selenite the starches of tubers showed a more varied play of colors than the cereal and leguminous starches which are produced above ground. On this fact Blyth has made another classification. Both are of value and interest.

Vogel's table of the different starches and arrowroots of commerce.

A.—Granules simple, bounded by rounded surfaces.

- I. Nucleus central, layers concentric.
  - *a.* Mostly round or from the side lens-shaped.
    - 1. Large granules .0396 – .0528$^{\text{mm}}$, *rye starch.*
    - 2. Large granules .0352 – .0396$^{\text{mm}}$, *wheat starch.*
    - 3. Large granules .0264, *barley starch.*
  - *b.* Egg-shaped, oval, kidney-shaped. Hilum often long and ragged.
    - 1. Large granules .032 – .079$^{\text{mm}}$, *leguminous starches.*
- II. Nucleus eccentric, layers plainly eccentric or meniscus shaped.
  - *a.* Granules not at all or only slightly flattened.
    - 1. Nucleus mostly at the smaller end .06 – .10$^{\text{mm}}$, *potato starch.*
    - 2. Nucleus mostly at the broader end or towards the middle in simple granules .022 – .060$^{\text{mm}}$, *maranta starch.*
  - *b.* Granules more or less strongly flattened.
    - 1. Many drawn out to a short point at one end.
      - *a.* At most .060$^{\text{mm}}$ long, *curcuma starch.*
      - *b.* As much as .132$^{\text{mm}}$ long, *canna starch.*
    - 2. Many lengthened to bean-shaped, disk-shaped, or flattened; nucleus near the broader end .044 – .075$^{\text{mm}}$, *banana starch.*
    - 3. Many strongly kidney-shaped; nucleus near the edge .048 – .056$^{\text{mm}}$, *sisyrinchium starch.*
    - 4. Egg-shaped; at one end reduced to a wedge, at the other enlarged; nucleus at smaller end .05 – .07$^{\text{mm}}$, *yam starch.*

B.—Granules simple or compound, single granules or parts of granules, either bounded entirely by plane surfaces, many angled, or by partly round surfaces.

- I. Granules entirely angular.
  - 1. Many with prominent nucleus. At most .0066$^{\text{mm}}$, *rice starch.*
  - 2. Without a nucleus. The largest .0088$^{\text{mm}}$, *millet starch.*
- II. Among the many angled also rounded forms.
  - *a.* No partly rounded forms present, angular form predominating.
    - 1. Without nucleus or depression very small, .0044$^{\text{mm}}$, *oat starch.*
    - 2. With nucleus or depression .0132 – .0220$^{\text{mm}}$.
      - *a.* Nucleus or its depression considerably rounded; here and there the granules united into differently formed groups, *buckwheat starch.*
      - *b.* Nucleus mostly radiatory or star-shaped; all the granules free, *maize (corn) starch.*
  - *b.* More or less numerous kettle-drum and sugar-loaf like forms.
    - 1. Very numerous eccentric layers; the largest granules .022 – .0352$^{\text{mm}}$, *batata starch.*
    - 2. Without layers or rings .08 – .022$^{\text{mm}}$.
      - *a.* In the kettle drum-shaped granules the nucleal depression mostly widened on the flattened side, .008 – .022$^{\text{mm}}$, *cassava starch.*
      - *b.* Depression wanting or not enlarged.
        - *aa.* Nucleus small, eccentric, .008 – .016$^{\text{mm}}$, *pachyrhizus starch.*
        - *bb.* Nucleus small, central, or wanting.
          - *aaa.* Many irregular angular forms .008 – .0176$^{\text{mm}}$, *sechium starch.*
          - *bbb.* But few angular forms; some with radiatory nucleal fissure, .008 – .0176, *castanospermum starch.*

C.—Granules simple and compounds, predominant forms, egg form and oval, with eccentric nucleus and numerous layers, the compound granule made up of a large granule and one or more relatively small kettledrum-shaped ones, .025 – .066^{mm}, *sago starch.*

Muter's table for the detection of starches when magnified about 230 diameters.[*]

[All measurements are given in decimals of an inch.]

Group I: All more or less oval in shape and having both *hilum* and rings visible.

Name.	Shape.	Normal measurements.	Remarks.
Tous les mois	Oval, with flat ends	.00370 to .00185	Hilum annular, near one end and incomplete rings.
Potato	Oval	.00270 .00148	Hilum annular, rings incomplete, shape and size very variable.
Bermuda arrowroot	Sack-shaped	.00148 .00129	Hilum distinct annular, shape variable, rings faint.
St. Vincent arrowroot	Oval-oblong	.00148 .00129	Hilum semilunar, rings faint, shape not very variable.
Natal arrowroot	Broadly ovate	.00148 .00129	Hilum annular, in center and well marked complete rings.
Galangal	Skittle-shaped	†.00135	Hilum elongated, very faint incomplete rings.
Calumba	Broadly pear-shaped	†.00185	Hilum semilunar, faint but complete rings, shape variable.
Orris root	Elongated-oblong	†.00092	Hilum faint, shape characteristic.
Turmeric	Oval-oblong, conical	†.00148	Very strongly-marked incomplete rings.
Ginger	Shortly conical, with rounded angles.	†.00148	Hilum and rings scarcely visible, shape variable but characteristic.

Group II: With strongly-developed *hilum* more or less stellate.

Name.	Shape.	Normal measurements.	Remarks.
Bean	Oval-oblong	†.00135	Fairly uniform.
Pea	Like bean	.00111 —.00074	Very variable in size, with granules under .00111 preponderating.
Lentil	Like bean	†.00111	Hilum, a long depression seldom radiate.
Nutmeg	Rounded	†.00055	The small size and rounded form distinctive.
Dari	Elongated hexagon	†.00074	Irregular appearance and great convexity distinctive.
Maize	Round and polygonal	†.00074	The rounded angles of the polygonal granules distinctive.

Group III: Hilum and rings practically invisible.

Name.	Shape.	Normal measurements.	Remarks.
Wheat	Circular and flat	.00185 to .00009	*Very variable* in size and *very dull* polarization in water.
Barley	*Slightly* angular circles	‡.00073	The majority measuring about .00073 distinctive.
Rye	Like barley	.00148 .00009	Small granules, quite round, and here and there cracked.
Jalap	Like wheat		Polarizes *brightly* in water.
Rhubarb	do	.00055 §.00033	Polarizes between jalap and wheat, and runs smaller and more convex.
Senega	do	.00148 —.00009	Measurements the only guide.
Bay berry	do	.00074 —.00011	
Sumbul	do	.00074 —.00009	

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[*] Analyst 1, 172–174, November 15, 1876.

† About.

‡ And a few four times this size.

§ For small granules.

Muter's table for the detection of starches, &c.—Continued.

Group III: Hilum and rings practically invisible—Continued.

Name.	Shape.	Normal measurement.	Remarks.
Chestnut	Very variable	.00090 — .00009	Variable form, and small but regular size, distinctive.
Acorn	Round-oval	*.00074	Small and uniform size, distinctive.
Calabar bean	Oval-oblong	.00296 .00180	Large size and shape characteristic.
Liquorice	Elongated-oval	*.00018	Small size and shape distinctive.
Hellebore (green or black)	Perfectly rotund	.00037 .00009	Small, regular size and rotundity, distinctive.
Hellebore (white)	Irregular	.00055 .00009	Irregular shape and faint central depression, distinctive.

Group IV: More or less truncated at one end.

Name.	Shape.	Normal measurement.	Remarks.
Cassia	Round	.00111 to .00018	Round or muller shaped granules and faint circular hilum.
Cinnamon	Like cassia	.00074 .00009	More frequently truncated than cassia, and smaller.
Sago (raw)	Oval-ovate	.00260 .00111	Has circular hilum at convex end and rings faintly visible.
Sago (prepared)	......do	.00260 .00111	Has a large oval or circular depression, covering one-third nearly of each granule.
Tapioca	Roundish	.00074 .00055	A little over 50 per cent. truncated by *one* facet, and a pearly *hilum*.
Arum	Like tapioca	*.00056	Smaller than tapioca and truncated by *two* facets.
Belladonna	......do	....	Not distinguishable from tapioca.
Colchicum	......do	*.00074	Larger than tapioca, and contains many more truncated granules.
Scammony	......do	*.00045	Smaller than tapioca, more irregular, and *hilum* not visible.
Canella	Very variable	.00033 — .00022	Very variable, form and small size the only points.
Podophyllum	Like tapioca	*.00040	Like scammony, but has visible *hilum* in most of the granules.
Aconite	......do	*.00037	Like tapioca, but half the size.

Group V: All granules more or less polygonal.

Name.	Shape.	Normal measurement.	Remarks.
Tacca	Poly or hexagonal	.00075 to .00037	Distinguished from maize by its *sharp* angles.
Oat	Polygonal	*.00037	Larger than rice and hilum visible in some granules.
Rice	......do	.00030 — .00020	Measurement using one-eighth or one-twelfth inch-power, and then hilum visible.
Pepper	......do	.00020 — .00002	Do.
Ipecacuanha	......do	*.00018	Some round and truncated granules, adhering in groups of three.

*About.

Blyth's classification.

DIVISION I.—Starches showing a play of colors with polarized light and selenite plate.

Class I. The hilum and concentric rings clearly visible, all the starches oval or ovate, including tous les mois, potato, arrowroot, calumba, orris root, ginger, galangal, and turmeric.

DIVISION II.—Starches showing no iridescence, or scarcely any, when examined by polarized light and selenite.

Class II. The concentric rings all but invisible, hilum stellate, including bean, pea, maize, lentil, dari, and nutmeg.

Class III. Starches having both the concentric rings and hilum invisible in the majority of granules. This important class includes wheat, barley, rye, chestnut, acorn, and many starches in medicinal plants.

Class IV. All the granules truncated at one end. This class includes sago, tapioca and arum, several drugs, and cinnamon and cassia.

Class V. In this class all the granules are angular in form and it includes oats, tacca, rice, pepper, as well as ipecacuanha starch.

Of the starches which are included in the preceding classification but a limited number will be met with in spices and their adulterants or in the commoner foods. One must, however, be able to readily recognize the following:

Starches natural to spices and condiments.	Starches of admixture.
Ginger.	Wheat and other cereals.
Pepper.	Corn.
Nutmeg.	Oats.
Cassia.	Barley.
Pimento.	Potato.
Cinnamon.	Maranta and other arrowroots.
Cayenne.	Rice.
	Bean.
	Pea.
	Sago.
	Buckwheat.

The remainder may be found in other foods and in drugs and cannot well be omitted therefore from our classifications.

No one of these is complete in itself, but from the characters given and with the aid of our illustrations the starches which commonly occur in the substances which are here considered may usually be identified without difficulty.

In practice the manipulation of the microscope and the preparation of the object requires some little experience, but not more than analysts usually have had. For the benefit of those who have had none, it may be said that a small portion of the starch or spice is taken up upon a clean camel's hair brush and dusted upon a common slide. The excess is blown away and what remains moistened with a drop of a mixture of equal parts of glycerine and water or glycerine and camphor-water and covered with a cover glass. It is well to have a small supply of the common starches in a series of tubes, which can be mounted at any moment and used for comparison. They can be permanently mounted by making with cork borers of two sizes a wax cell-ring equal to the diameter of the cover glass, and after cementing the cell to the slide with copal varnish thinned with turpentine and introducing the starch and glycerine mixture, fixing the cover glass on after running some of the cement over the top of the ring. A little experience will enable one to put the right amount of liquid in the cell and to make a preparation which will keep for some time. After several months, however, it is difficult to distinguish the rings which mark the development of the granule, and although for reference as to size and form the preparation is satisfactory, it is always advisable in doubtful cases to examine some fresh material.

For other purposes the starches should be mounted in prepared Canada balsam or dammar by well-known methods. In this medium they can be preserved indefinitely, but are scarcely visible with ordinary illumination, and must be viewed by polarized light, which brings out distinctive characters, not seen as well or at all in other mounts.

Appearance in glycerine and water.—When mounted in the manner already described, or in water alone if for only temporary use, and examined under a microscope with an objective of equivalent focus of one-half to one-fifth inch, and with means for oblique illumination, the starches will display the characteristics which have been mentioned, and which are illustrated on Plates 26, 27, and 28. These illustrations have been drawn from nature by Dr. George Marx, and represent the starches as nearly as possible, as they are seen, and not as in many of the absurd illustrations of the handbooks of microscopists of the past and present day, which are entirely ideal, representing the granules not as extremely translucent bodies, but with the rings or layers as strongly-marked lines. Examined in this manner the size, shape, presence or absence of a nucleus or hilum, and of the rings and their arrangement, can be made out, and the starch referred to its proper position.

Appearance in balsam with polarized light.—Mounted in balsam the starches are scarcely visible under any form of illumination with ordinary light, the index of refraction of the granules and the balsam being so nearly alike. When, however, polarized light is used the effect is a striking one, and is illustrated in Plates 19 to 21. It is very easy to distinguish all the characteristics, except the rings, the center of the cross being at the nucleus of the granule.

With the selenite plate a play of colors is produced, which is peculiar to some of the starches and forms the basis of Blyth's classification.

The principal starches which are met with may be described as follows, in connection with our illustrations, beginning with those of the arrowroot class, including the potato, ginger, and turmeric.

Potato starch.—The starch grains of the potato are very variable in size, being found from .05 to .10^{mm} in length, and in shape from oval and allied forms to irregular and even round in the smallest. These variations are illustrated in Fig. 57, but the frequency of the smaller granules is not as evident as in Figs. 30 and 31. The layers are visible in some granules with great distinctness and in others hardly at all, being rather more prominent in the starch as obtained from a freshly cut surface. The rings are more distinct, too, near the hilum or nucleus, which in this, as in all tuberous starches, is eccentric, shading off toward the broader or more expanded portion of the granule. The hilum appears as a shadowy depression (Fig. 57) and with polarized light its position is well marked by the junction of the arms of the cross, and it will be found by comparison of Figs. 31 and 32, on Plate XVI, that in the potato it is oftener at the smaller end of the granule and in the arrowroot at the larger. With polarized light and a selenite plate the beautiful play of colors is obtained which is the basis of Blyth's classification. The smaller granules, which are nearly round, may readily be confused with other starches, but their presence serves at once to distinguish this from *Maranta* or *Bermuda arrowroot* starch.

Rarely compound granules are found composed of two or three single ones each with its own nuclus.

Of the same type as the potato starch are the various arrowroots, the only one of which commonly met with in this country being the Bermuda, the starch of the rhizome of *Maranta arundinacea*, and the starch of Turmeric.

Maranta starch.—The granules are usually not so varied in size or shape as those of the potato, as may be seen in Figs. 30, 31, and 32, averaging about .07$^{\text{mm}}$ in length. They are about the same size as the average of the latter, but are never found as large or as small, which, together with the fact that the end at which the nucleus appears is broader in the Maranta and more pointed in the potato, enables one to distinguish the starches without difficulty. With polarized light the results are similar to those seen with potato starch, and this is a ready means of distinguishing the two varieties, by displaying in a striking way the form of the granule and position of the hilum, as is illustrated in Figs. 31 and 32.

Curcuma or turmeric starch.—Tumeric contains a starch (Fig. 63, Plate XXVIII), which, although of the arrowroot class, is quite distinct in appearance from those which we have described. It is most irregular in outline, so that it is impossible to define its shape or to do more than refer to the illustration. Many of the granules are long and narrow and drawn out to quite a point. The rings are distinct in the larger ones. The size is about that of the Maranta.

Ginger starch (Figs. 41 and 42, Plate XXI, and Fig. 58, Plate XXVII).—This starch is of the same class as those from the potato and Maranta and several others which are of under-ground origin. In outline it is not oval like those named, but more rectangular, having more obtuse angles in the larger granules and being cylindrical or circular in outline in the smaller.

It averages nearly the same size as Maranta starch, but is much more variable both in size and form. The rings are scarcely visible even with the most favorable illumination.

Sago starch.—This exists in two modifications in the market; as raw and as prepared sago. In the prepared condition it is characterized by a larger circular depression in the center of most of the granules. The rings are not visible. They are mostly circular in form or approaching it, and vary from .025 to .065^{mm} in diameter.

Leguminous starches, pea and bean (Figs. 39 and 40, Plate XX, and Figs. 60 and 61, Plate XXVII).—These starches produce but a slight effect with polarized light. The rings are scarcely visible, and the hilum is stellate or much cracked along a median line; the bean more so than the pea, the latter resembling fresh dough kneaded again into the center as in making rolls, and the former the shape assumed by the same after baking. They are both somewhat variable in size, ranging from .025 to .10^{mm} in length.

Nutmeg starch (Fig. 64, Plate XXVIII).—This starch, which in some respects resembles the preceding—the rings being scarcely visible and not iridescent with polarized light—is much smaller in size and quite variable. The larger granules are at times as long at .05^{mm} and the smallest smaller than .005^{mm}. They are of extremely irregular form, with angular depressions and angular outlines, and are distinguished by a budded appearance, caused by the adherence of small granules to the larger.

Capsicum starch (Fig. 67, Plate XXVIII) is nearly circular or rounded, polyhedral in form, with scarcely visible rings, and in most cases a depressed hilum resembling in size and shape corn starch, but having peculiar irregularities, which distinguish it, such as a rosette-like formation on a flattened granule or a round depression at one end. It does not polarize as actively as maize starch, and can be distinguished from rice by the greater angularity of the latter.

Pepper starch (Fig. 65, Plate XXVIII) is the most minute starch that is usually met with, not averaging over .001^{mm}, nor exceeding .005. It is irregularly polyhedral, polarizes well, but requires a high power to discover any detail when a hilum is found. It cannot be confused with other starches.

Cinnamon starch (Fig. 46, Plate XXIV, and Fig. 66, Plate XXVIII) has an extremely irregular, polyhedral or distorted granule, often united in groups with smaller granules adherent to the larger ones. In size it varies from .001 to .025, averaging nearly the latter size. In some granules a hilum can be distinguished, but no rings. It is readily detected with polarized light.

Buckwheat starch (Fig. 62, Plate XXVIII) is very characteristic. It consists of chains or groups of angular granules, with a not very evident circular nucleus and without rings. The outline is strikingly angular and the size not very variable, being about .01 to .015.

Maize or corn starch (Fig. 33, Plate XVII, and Fig. 54, Plate XXVI).—The granules of corn-starch are largely of the same size, from .02 to .03^{mm} in diameter, with now and then a few which are much smaller. They are mostly circular in shape or rather polyhedral, with rounded angles. They form very brilliant objects with polarized light, but with ordinary illumination show but the faintest sign of rings, and a well-developed hilum, at times star-shaped, and at others more like a circular depression.

Rice starch (Figs. 35 and 36, Plate XVIII, and Fig. 55, Plate XXVI) is very similar to corn starch, and easily confused with it, being about the same size. It is, however, distinguished from it by its polygonal form, and its well-defined angles. The hilum is more prominent and more often stellate or linear. Several granules are at times united.

Wheat starch (Fig. 34, Plate XVII, and Fig. 50, Plate XXVI) is quite variable in size, varying from .05 to .012^{mm} in diameter. It belongs to the same class as barley and rye, the hilum being invisible and the rings not prominent. The granules are circular disks in form, and there are now and then contorted depressions resembling those in pea starch. It is the least regular of the three starches named and does not polarize actively.

Barley starch (Fig. 37, Plate XIX, and Fig. 51, Plate XXVI) is quite similar to that of wheat, but does not vary so much in size, averaging .05^{mm}; has rings which are much more distinct, and very small granules adhering to the largest in bud-like forms.

Rye starch (Fig. 52, Plate XXVI) is more variable in size, many of the granules not exceeding .02^{mm}, while the largest reach .06 to .07^{mm}. It lacks distinctive characteristics entirely, and is the most simple in form of all the starches we have described.

Oat starch (Fig. 38, Plate XIX, and Fig. 53, Plate XXVI) is unique, being composed of large compound masses of polyhedral granules from .12 to .02^{mm} in length, the single granules averaging .02 to .015 mm. It does not polarize actively as may be seen in figure and plate, and displays neither rings nor hilum. The illustration shows its nature with accuracy.

Our descriptions, it will be seen, do not agree entirely with those of other authors, which in the same way do not agree among themselves. This shows a variation in the peculiarities of size, shape, &c., which must be carefully allowed for, and the necessity for every investigator to compare a starch which he is desirous of identifying with authentic specimens.

STRUCTURE AND PECULIARITIES OF THE COMMONER ADULTERANTS.

Before proceeding to the consideration of the normal structure and composition of the spices and condiments and the adulterations detected in commercial specimens, it is well to become familiar with the characteristics of the common adulterants and materials which are liable to be used for this purpose.

The starches have been already described and their value as a means of identifying different vegetable materials noted. By this means we are able to detect the different cereals which are often added as diluents.

Maize, or corn as it is commonly known, is a common adulterant. By selecting particles from the ground material and crushing them the character of the starch may be recognized, but in a cursory examination the first sign of the presence of this cereal is the discovery of one of the thin outer coats of the grain which becomes detached in milling and, being tough, is not readily reduced. In yellow corn it has a peculiar pinkish color and simple structure of longitudinal cells. One should learn to recognize it from a specimen ground for the purpose.

Rice, which in its broken unmarketable form is sometimes used as a diluent, may be recognized by the brilliant appearance of the hard white particles which must be picked out of the spice under a hand lens, crushed, and examined as usual. Rice bran has not been met with.

The two cereals named are the only ones which are commonly met with which introduce starch. Rarely clean *wheat bran* is added, which can be recognized by its distinctive structural character, illustrated in many hand-books, but which can be learned much better from an authentic specimen, which should be soaked in chloral hydrate.

As modified cereals, we find refuse bread, cracker-dust, and ship-bread in which the wheat starch is much changed from its original form by the heat and moisture of the cooking process so that at times it might be confused with a leguminous starch. The softness of the particles and the ease with which they fall to pieces in water reveals the nature of the material. It is a common diluent.

Oil-seed, oil-cake, and husks are very commonly used in many parts of the country for purposes of sophistication. They are most readily recognized by the peculiar structure of the outer coats of the seed. The particles which can usually be found and selected with a dissecting microscope should be examined in alcohol or glycerine, or a mixture of the two, as the outer coats of some seeds, such as mustard, are swollen by water and become indistinct. The appearance of mustard hulls is given on page 172, and the many varieties of the cruciferous seeds resemble it much, so that it is difficult to distinguish them, which is, however, not important. They are generally distinguished by the outer layer of hexagonal cells, and a middle and an inner coating which consist of peculiar angular cells, the latter much larger than the former, which are the most characteristic, and should be compared with specimens of seed of known origin. The structure of some of them is diagramatically presented in fig. 6, from Schimper. After soaking in chloral hydrate the remaining interior layers are perhaps more easily made out, and in some cases after moderate bleaching with nitric acid and chlorate. The interior of these seeds is not blued by iodine.

Peanut or groundnut cake is recognized by the characteristic structure of the red-brown coat which surrounds the seed, which consists of polygonal cells with peculiar saw-toothed thickening of the walls. The seed itself consists of polygonal cells, full of oil and starch granules, which are globular in form and not easily confused with pepper starch.

The structure of the brown membrane is best made out in chloral hydrate, which removes the red color and leaves the fragments of a bright yellow.

Linseed cake distinguished by the fact that its husk is made up of one or two characteristic elements. The outer coat or epiderimis is colorless and swells up in water, forming a mucilage like the mustard seed. Beneath this is a layer of thin round yellow cells, while the third is very characteristic, and consists of narrow, very thick-walled dotted vessels. Next to these is an inner layer of compact polygonal cells, with fairly thin but still thickly dotted white walls and dark-brown contents, containing tannin. The endosperm and embryo are free from starch; nor are they colored yellow by potash, as is the case with mustard and rape cake.

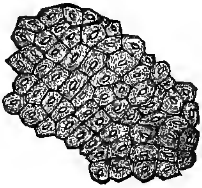

Fig. 6.

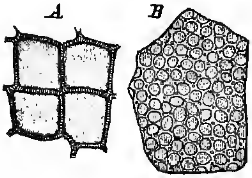

Fig. 7.

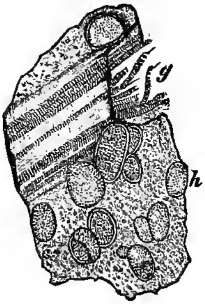

Fig. 8.

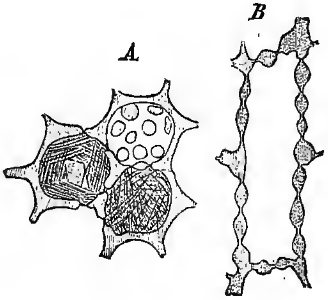

Fig. 9.

Fig. 6. Rape-seed husk (exterior surface).
Fig. 7. Linseed kusk. *A* more, *B* less magnified.
Fig. 8. Almond shell fragment. *h* hairs, *g* spiral vessels. × 70.
Fig. 9. Palm-seed. Interior. × 240. (After Schimper.)

Palm-cake is probably not common enough in this country to be used as an adulterant; nor are *olive stones*, which as they consist, after bleaching with Schulze's reagent, almost entirely of very thick stone cells, are easily made out under polarized light.

Cocoanut shells are often used, it seems from the evidence of the Canadian analysts. They are similar to the olive stones in structure but more complicated, as in addition to the numerous short stone cells there are many long ones with thinner walls, and here and there spiral vessels, from the fibrous tissue, all of which are only readily seen after bleaching. When the shells are roasted or charred they refuse to bleach, and it is then only possible to class the particles on which the reagents do not act as roasted shells or charcoal. They are frequently used in peppers to give color to material rendered too light by white adulterants.

The composition of these substances is shown in the following determination which reveal the effect of their addition upon the normal composition of the spices:

	Olive stones.	Cocoanut shells.
Water	5.63	6.15
Ash	4.28	2.13
Fiber	41.33	37.15
Albuminoids	1.56	1.25
Nitrogen	.25	.20

More in regard to olive stones will be learned when the discussion of the adulteration of pepper is considered.

Buckwheat hulls after bleaching with Schulze's reagent show a preponderance of tissue made up of long, slender, and pointed sclerenchyma cells and a smaller amount of reticulated tissue resembling the cereals somewhat and cayenne pepper. Portions of the endosperm or interior of the seed are also visible, and consist of an agglomeration of small hexagonal cells which originally contained starch. The starch is readily recognized by its peculiar characteristics. The sclerenchyma is, of course, optically active and forms a beautiful and distinctive object with polarized light.

Sawdust of various woods may be recognized by the fragments of various spiral and dotted vessels and fibrous material which are not found in the spices or other adulterants.

Bark, in some parts of the world a common addition to pepper, is detected by its stone cells, which are larger than those of pepper, and of different form and more numerous, and by its fibrous vessels which are made out readily after bleaching. The source of a particular bark cannot, however, be made out.

Rice bran.—This, as several other similar products, is made up prominently of two series of cells at right angles to each other, which make up the outer coats of the grain. The structure is best made out after soaking in chloral-hydrate. The cells of one series are long, small, and thin-walled, and are arranged in parallel bundles. The others have very much thickened walls, and are only two or three times as long as broad. They are at times distinguished, for convenience, as the longitudinal and transverse cells. The remaining layers of the bran are not prominent.

Clove stems, used frequently as a diluent, can be distinguished by their peculiar yellow-dotted vessels and their large and quite numerous stone-cells, neither of which are seen prominently in the substances which are adulterated.

These suggestions of the peculiarites of the different adulterants should, of course, be confirmed and supplemented, and the eye accustomed to recognize their structure by means of a study of the actual substances, which should always be at hand in the dry and ground condition for reference. Quickness and certainty will be much advanced by such facilities.

Reference may also be fittingly made here to chemical operations of a general nature, which are applicable to all the spices.

CHEMICAL EXAMINATION.

Determinations of a quantitative nature should include—

Water.—A portion of the powdered spice which should pass a 60-mesh sieve, one gram, is to be dried at 105° to 110° C. in an air bath, provided with a regulator, until on successive weighings a gain is found showing that oxidation has begun. Twelve hours, or overnight, usually suffices. The loss is water, together with the largest part of the volatile oil. Deduction of the volatile oil, as determined in the ether extract, will give a close approximation to the water.

Ash.—In the same portion the ash is determined by incineration at a very low temperature, such as may be best obtained in a gas muffle, which is a most convenient arrangement for work of this description, and far superior to any kind of lamp or burner. The proportion of ash insoluble in acid may also be determined where there is reason to believe that *sand is present.*

Volatile oil and ether extract.—Two grams of the substance are extracted for twenty hours in a siphoning extraction apparatus on the Soxhlet principle, with Squibbs's best ether. The apparatus we use in this laboratory, arranged by Mr. A. E. Knorr, he describes as follows:

The substance under examination is placed in a test tube, which is then inserted into a continuous extraction apparatus of the intermittent siphon class. The tubes used for this purpose are ordinary test tubes, the bottom of which has been blown out. A wad of washed cotton of sufficient thickness is put into the lower end of the tubes to prevent any solid particles of the sample from finding their way into the receiving flask; another wad of cotton is packed on top of the sample, and the apparatus is then so adjusted that the condensed ether drops into the tube, and, percolating through the sample, siphons into the receiving flask, when the operation is continued *ad infinitum.*

It is absolutely necessary to use the best Squibbs's ether in order to avoid extracting substances other than oil and soluble in alcohol, and to continue the extraction for at least the time named, as piperine and several other proximate principles are not extremely soluble in ether. If these precautions are followed we have found no difficulty in extracting all the piperine, for example, and obtaining duplicate results of great accuracy. On stopping the extraction the extract is washed into a light weighed glass dish, and the ether allowed to evaporate spontaneously and not too rapidly, as in the latter case water is condensed in the dish, which it is difficult to remove. When the ether has disappeared—which ought not to take too long, as in that case some oil is volatilized—the dish is placed in a large dessicator, with pumice and sulphuric acid—chloride of calcium having been shown to be useless*—and allowed to remain overnight, to remove any moisture. The loss of oil by this process is hardly appreciable. The dish is then weighed, and afterwards heated to 110° C. for some hours, to drive off the volatile oil, beginning at a rather low temperature, as the oil is easily oxidized, and then is not volatile. The residue is weighed, the difference being calculated to volatile oil, and then examined as to its composition of purity. The results are fairly satisfactory, as appears from the following duplicates:

Duplicate fat and volatile oil determinations in spices.

No.	Per cent.	Per cent.	No.	Per cent.	Per cent.
4629.....	5. 49	8. 38	4632.....	4. 11	3. 66
4629.....	5. 23	8. 51	4632.....	3. 93	4. 22
4630.....	5. 15	6. 54	4633.....	5. 06	6. 94
4630.....	4. 72	6. 50	4633....	5. 33	7. 68
4631.....	6. 64	13. 19	4634.....	5. 15	3. 50
4631.....	6. 84	12. 14	4634.....	2. 26	3. 59

Alcohol extract.—This may be made in the same manner as the ether extract, using, of course, the substance already extracted. The solvent may be either absolute alcohol, that of 95 per cent. by volume, or 80 per cent. by weight. The latter is preferable in most cases, as there is no definite point with the stronger spirit at which the extraction is complete.

In the investigation of spices merely for adulteration this extraction is of little value.

Starch, &c., reducing sugars.—The amount of reducing material produced by boiling the spices with dilute acid serves with several as an index of purity. In the case of pepper, which contains naturally a large amount of starch, the addition of the common fibrous adulterants reduces the equivalent of reducing sugars which are indicated by Fehling's solution after boiling with acid. Lenz and several others have examined the value of this determination. The conclusions which are deducible from their experience and our own are that with attention to proper condition to insure complete conversion to dextrose the results are of value, though apt to fall out too low. It has been found desirable in our laboratory to run as a check a parallel determination on a substance of known reduction equivalent. In this way any variation may be detected.

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* Fleischer. E., 1884, Zeit. Anal. Chem., **23**, 33.

The method of Lenz is described under pepper and its composition. The conditions which we have found most desirable are as follows:

The 2 to 5 grams of the material, usually 4, which should pass an 80-mesh sieve, must be extracted with strong alcohol and with cold water for some time to remove substances not starch which might be acted on by the acid or reduce Fehling's solution. Then, without drying, it is washed off the filter into an Erlenmeyer flask with about 175 c. c. of water and enough strong C. P. hydrochloric acid added, about 25 c. c., to make the liquid 4 to 5 per cent. of acid gas. The flask is then supplied with a condenser and boiled for four hours, or the liquid may be put in a patent rubber-stoppered beer bottle and digested in a steam bath; but the latter method is not as certain. After the boiling and cooling, the residue is filtered out, the liquid accurately neutralized with sodic hydrate and made up to 500 c. c. It is then titrated as usually, and can be calculated to glucose or starch on any basis desired. Thorough previous extraction and uninterrupted boiling are the two most important conditions. Without extraction the results are most uncertain and unreliable.

Determination of tannin.—The amount of tannin in certain spices, such as cloves and allspice, is quite constant when they are of good quality. Dr. Ellis of Toronto has therefore recommended this as a good means of detecting adulteration in these spices. He has published as yet nothing in regard to his methods of application. In our experiments it has been found to be of some value, but that it is hardly worth while as a mere aid to the detection of adulteration to go so far as the actual determination of tannin, it being quite sufficient to determine the amount of material oxidizable with permanganate which is extracted by water after the careful removal of the oil, &c., by ether. This avoids the tedious use of hide powder, or glue, and furnishes results which are relatively of the same value.

The analyst should therefore prepare himself for carrying out the first part of the modified Löwenthal process, as it is described in Sutton,* and more elaborately in the Berichte über die Verhandlungen der Commission zur Bestellung einer einheitliche Methode der Gerbstoffbestimmung, and in Annales de la Science agronomique, Tome 1, 1886.

Particular pains should be taken to secure a good article of indigo carmine, as without this the results are unreliable. That recommended by Schröder is manufactured by Gehe & Co., of Dresden, and is known as *Carminum cœruleum.* It should be imported for use.

Without going into particulars as to the reagents employed in the process, which are probably familiar to all, a few words of caution as to detail of manipulation will be of value.

As has been said, preliminary extraction of the material with Squibbs best ether is necessary to remove oil and other substances not tannin, on which the permanganate may act. Ordinary ether will not answer,

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* Sutton, Volumetric Analysis, 3d edition, pp. 276.

as it contains so much alcohol and water as to dissolve some of the tannin. The substance freed from ether should be extracted with boiling water, and the extract made up to such dilution that 10 c. c. is equal to about 10 c. c. of the thirtieth normal permanganate solution used. The titration must be performed slowly to insure accuracy, the permanganate being run in at a rate of not more than a drop in a second or three in two seconds. The eye must become accustomed to the bleaching of the indigo and select some one tint of yellow as the end of reaction. It is then possible to obtain duplicates agreeing within .1 c. c. even on entirely different tests, as the following figures show:

Serial No.	C. c. of Perm.	Per ct. of tannin.
4904.....	4. 33	22. 46
	4. 3	22. 36
	4. 35	22. 62
	4. 15	21. 58

The results may be calculated to oxygen consumed or to percentage of quercitannic acid, which would not be strictly correct, 1 c. c. of $\frac{N}{30}$ permanganate being equivalent to .0052 grams of quercitannic acid. The results obtained with cloves and allspice will be found under those spices.

Crude fiber.—This is a merely relative determination, as the term crude fiber designates nothing absolute beyond the fact that a certain amount of substance is insoluble in acid and alkali of certain strength after treatment for a definite length of time at a definite temperature. The conditions selected by us are, 2 grams of substance, 200 c. c. of 5 per cent. hydrochloric acid, steam bath two hours, raising the liquid to a temperature of 90° to 95° C., filration on linen cloth, washing back into beaker with 200 c. c. 5 per cent. sodic hydrate, steam bath two hours, filtration on asbestos, washing with hot water, alcohol, and ether, drying at 120° weighing, ignition and crude fiber from loss in weight. This method agrees practically with that known as the Weende method, and while furnishing results which are of some comparative value, leaves much to be desired. The subject will probably be reviewed soon by the Association of Official Agricultural Chemists of the United States.

Nitrogen and albuminoids.—The methods of determining nitrogen and albuminoids have been discussed and described at length in Bulletin No. 12 of this division. The details of the method of Kjeldahl, as given by Dr. Jenkins, which is the most convenient, are as follows:

Determination of nitrogen by the method of Kjeldahl.

REAGENTS AND APPARATUS.

(1) Hydrochloric acid whose absolute strength has been determined, (a) by precipitating with silver nitrate and weighing the silver chloride, (b) by sodium carbonate, as described in Fresenius's Quantitative Analysis, second American edition, page 680,

and (c) by determining the amount neutralized by the distillate from a weighed quantity of pure ammonium chloride boiled with an excess of sodium hydrate.

(2) Standard ammonia whose strength, relative to the acid, has been accurately determined.

(3) "C. P." sulphuric acid, Sp. Gr. 1.83, free from nitrates and also from ammonium sulphate, which is sometimes added in the process of manufacture to destroy oxides of nitrogen.

(4) Mercuric oxide, HgO, prepared in the wet way. That prepared from mercury nitrate cannot safely be used.

(5) Potassium permanganate tolerably finely pulverized.

(6) Granulated zinc.

(7) A solution of 40 grams of commercial potassium sulphide in one liter of water.

(8) A saturated solution of sodium hydrate free from nitrates, which are sometimes added in the process of manufacture to destroy organic matter and improve the color of the product.

(9) Solution of cochineal prepared according to Fresenius's Quantitative Analysis, second American edition, page 679.

(10) Burettes should be calibrated in all cases by the user.

(11) Digestion flasks of hard, moderately thick, well-annealed glass. These flasks are about 9 inches long, with a round, pear-shaped bottom, having a maximum diameter of 2½ inches, and tapering out gradually in a long neck, which is three-fourth of an inch in diameter at the narrowest part, and flared a little at the edge. The total capacity is 225 to 250 cubic centimeters.

(12) Distillation flasks of ordinary shape, 550 cubic centimeters' capacity, and fitted with a rubber stopper and a bulb tube above to prevent the possibility of sodium hydrate being carried over mechanically during distillation. This is adjusted to the tube of the condenser by a rubber tube.

(13) A condenser. Several forms have been described, no one of which is equally convenient for all laboratories. The essential thing is that the tube which carries the steam to be condensed shall be of block tin. All kinds of glass are decomposed by steam and ammonia vapor, and will give up alkali enough to impair accuracy. (See Kreussler and Henzold, Ber. Berichte, *XVII*, 34.) The condenser in use in the laboratory of the Conn. Exp. Station, devised by Professor Johnson, consists of a copper tank supported by a wooden frame, so that its bottom is 11 inches above the work-bench on which it stands. This tank is 16 inches high, 32 inches long, and 3 inches wide from front to back, widening above to 6 inches. It is provided with a water-supply tube which goes to the bottom and a larger overflow pipe above. The block-tin condensing tubes, whose external diameter is ⅜ of an inch, 7 in number, enter the tank through holes in the front side of it near the top, above the level of the overflow, and pass down perpendicularly through the tank and out through rubber stoppers tightly fitted into holes in the bottom. They project about 1½ inches below the bottom of the tank, and are connected by short rubber tubes with glass bulb tubes of the usual shape, which dip into glass precipitating beakers. These beakers are 6½ inches high, 3 inches in diameter below, somewhat narrower above, and of about 500 cubic centimeters capacity. The titration can be made directly in them. The seven distillation flasks are supported on a sheet-iron shelf attached to the wooden frame that supports the tank in front of the latter. Where each flask is to stand a circular hole is cut, with three projecting lips, which support the wire gauze under the flask, and three other lips which hold the flask in place and prevent its moving laterally out of place while distillation is going on. Below this sheet-iron shelf is a metal tube carrying seven Bunsen burners, each with a stop-cock like those of a gas combustion furnace. These burners are of larger diameter at the top, which prevents smoking when covered with fine gauze to prevent the flame from striking back.

(14) The stand for holding the digestion flasks consists of a pan of sheet-iron 29 inches long by 8 inches wide, on the front of which is fastened a shelf of sheet-iron

as long as the pan, 5 inches wide and 4 inches high. In this are cut six holes 1⅝ inches in diameter. At the back of the pan is a stout wire running lengthwise of the stand, 8 inches high, with a bend or depression opposite each hole in the shelf. The digestion flask rests with its lower part over a hole in the shelf and its neck in one of the depressions in the wire frame, which holds it securely in position. Heat is supplied by low Bunsen burners below the shelf. Dr. Jenkins has used asbestus paper under the flasks, but finds that with a little care the naked flame can be applied directly to the flask without danger.

THE DETERMINATION.

One gram of the substance to be analyzed is brought into a digestion flask with approximately 0.7 gram of mercuric oxide and 20 cubic centimeters of sulphuric acid. The flask is placed on the frame above described in an inclined position and heated below the boiling point of the acid for from five to fifteen minutes, or until frothing has ceased. The heat is then raised till the acid boils briskly. No further attention is required till the contents of the flask has become a clear liquid, which is colorless or at least has only a very pale straw color. The flask is then removed from the frame, held upright, and while still hot, potassium permanganate is dropped in carefully and in small quantity at a time till after shaking the liquid remains of a green or purple color. After cooling, the contents of the flask are transferred to the distilling flask with water, and to this 25 cubic centimeters of potassium sulphide solution are added, 50 cubic centimeters of the soda solution, or sufficient to make the reaction strongly alkaline, and a few pieces of granulated zinc. The flask is at once connected with the condenser and the contents of the flask are distilled till all ammonia has passed over into the standard acid contained in the precipitating flask previously described and the concentrated solution can no longer be safely boiled. This operation usually requires from twenty to forty minutes. The distillate is then titrated with standard ammonia.

The use of mercuric oxide in this operation greatly shortens the time necessary for digestion, which is rarely over an hour and a half in the case of substances most difficult to oxidize and is more commonly less than an hour. In most cases the use of potassium permanganate is quite unnecessary, but it is believed that in exceptional cases it is required for complete oxidation, and in view of the uncertainty it is always used. Potassium sulphide removes all mercury from solution and so prevents the formation of mercuro-ammonium compounds which are not completely decomposed by soda solution. The addition of zinc gives rise to an evolution of hydrogen and prevents violent bumping. Previous to use the reagents should be tested by a blank experiment with sugar, which will partially reduce any nitrates that are present which might otherwise escape notice.

This method cannot be used for the determination of nitrogen in substances which contain nitrates or certain albuminoids.

In case non-albuminoid nitrogen is to be determined reference can be made to Stutzer and Ladd.*

These methods of analysis are suitable to all the spices and have been used with them. They are nothing but general processes, and are dependent for their value on uniformity in the way they are carried out and the manner in which peculiarities of proximate composition in different spices are considered in drawing conclusions. Determinations of particular substances, such as piperine, require, however, modifications, which must be described when discussing the analyses of each spice.

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* Rept. anal. Chem., **5**, 162, 163; Abs. Ber., **19**, 1885; and Ladd Rept. of New York Agric. Exp. Sta., 1886.

MUSTARD.

Mustard of commerce is the seed, whole or ground, of several species of the genus *Brassica*, cruciferous plants which grow wild and are cultivated under very various conditions. The two common varieties are the black or brown mustard, which has a very small seed and furnishes the most aroma, and the white, which is two or three times as large, often used in the whole condition in pickles and ground, either by itself or oftener in mixture with the brown seed, for the purpose of obtaining the desirable qualities of both.

In the ground mustard is found only the interior of the seed and small portions of the husks which have escaped the operation of bolting, which is always employed to remove the coarse fragments. The presence of these particles from their characteristic structure enable us to recognize the source from which the flour is derived and to detect the use of the mustard hulls as adulterants of other food materials. The husk of white mustard is represented, after a drawing by Schimper, in Fig. 10.

The outer colorless epidermis consists of angular plates or hexagonal tabular cells with a center of different brilliancy. They swell up and become slimy in water and must therefore, be observed in glycerine. At the best it requires some manipulation to see it well, and it is far less prominent in the brown seed. The next coat, denominated the subepidermal, is not prominent and can only be seen at all easily in the white seed.

The third layer is an important one. In it is found the coloring matter of the brown seed, and its absence is the cause of the lack of color in the white variety. Fragments of this layer are common in ground mustard. It is distinguished by the thick or colorless brown cell walls and their irregular dotted appearance. Once examined it will be readily recognized under other circumstances, as, for example, when the hull is used as an adulterant of pepper.

Between this layer and the next are some unimportant and difficultly discernible cells carrying in the brown seed some color.

Within these comes the important layer denominated the inner tunic by Hassall and the plasma layer by Schimper. It separates readily from the other parts of the husk and is often found by itself in the ground mustard. As its contents are broken up by water or chloral hydrate, glycerine or oil must be used as a mounting medium. The cells of which the layer consists are large, and with their contents are similar to the embryous envelope or false gluten cells of wheat, to which they correspond. They are much alike in both white and brown mustard. These structures are diagramatically represented in Fig. 7.

From the character of the exterior layer and the lack of color in the third layer, as well as minor differences which are not describable, but will appear to the patient investigator, it is always possible to tell whether the flour of mustard is a mixture of the two varieties or from one alone. Mounts in chloral hydrate, to a certain degree, are useful for adding to the transparency of the substance. The interior of the seed is made up of small soft parenchyma cells containing the oil and other constituents of the mustard, but without any trace of starch. For this reason the presence of starch is a certain indication of the addition of some diluent of a farinaceous nature. Simple treatment with iodine will therefore reveal the presence of wheat flour, which is a common adulterant of this condiment. The white color of the flour of course reduces the yellow color of the mustard, and it is usual, therefore, to restore the tint by either *turmeric* or *Martin's yellow*.

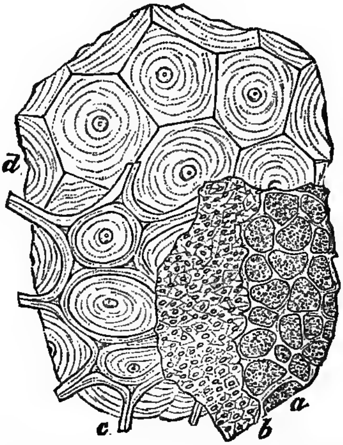

FIG. 10. Husk of white mustard. *a*, *b*, plasma layer; *c*, sub-epidermal; *d*, epidermis.

The former can be detected by a mechanical separation or a brown coloration with ammonia or by the peculiar color cells which it contains and the starch granules of the arrowroot class. The latter is not sufficient in amount to be confused with potato starch or that of flour. It has been already described.

Martin's yellow can be identified by extraction with cold 95 per cent. alcohol and examination, after evaporation of the solvent, as suggested by Waller and Martin, and this coloring matter seems to be often used, and cannot be pronounced as harmless as turmeric.

The substances mentioned are the common adulterants of mustard, in fact, so common that they have been accepted as necessary diluents, being considered desirable for toning down the pungency and adding to the keeping qualities of the ground material. Of late years, however, a reaction has taken place, and it is now possible to find brands of pure ground mustard.

In most of the samples which have come into our hands for examination flour and coloring matter are the only foreign substances which have been met with. From the investigations of foods chemists abroad, it would appear on the authority of Hassall and others that other species of mustard seed, rape seed, cayenne pepper, ginger, potato flour, rice, pea flour, seed meals, and several mineral substances are frequently found in the mustards of commerce, a conclusion which we have found justified by the presence in some of the lower-grade mustards which have come into our hands of yellow corn, ginger, mustard hulls, gypsum, and sand. The presence of these adulterants, which is only too common in the cheaper article when sold in bulk and under no brand, can be determined by mechanical means and by their structure, which is quite different from that of the mustard, and by the starches, which characterize some of them, as already explained.

While the adulterants of mustard, therefore, are, owing to the characteristic structure of the seed, easily detected with the microscope, in cases where there is doubt, or where further information is desired as to the probable proportion of diluent, recourse must be had to determinations of the chemical constituents of the sample.

CHEMISTRY OF MUSTARD.

Several investigators have made proximate analyses of mustard. Hassall has collected the following in regard to its composition, and has also made several analyses of pure and adulterated samples:

> Of these seeds no very complete quantatitive analyses have as yet been made, although many highly important particulars have been ascertained respecting their composition; thus black or brown mustard, as it is now generally named, consists for the most part of *fixed oil*, *myronic acid* $C_{10}H_{19}NS_2O_{10}$, which is combined with potash, forming a *myronate of potash*, and which acid is converted into the volatile oil of mustard or *sulphocyanide of allyl* C_4H_5 NS or $\left.\begin{matrix}CN\\C_3H_5\end{matrix}\right\}S$ through the agency of the *myrosin*, another constituent of brown mustard, when the two are brought into contact through the medium of water, *vegetable albumen*, a *bitter principle*, a little *gum* and *sugar*, *a peculiar green substance*, *cellulose* and *mineral matter*.
>
> White mustard differs essentially in its composition from brown; it also contains *fixed oil*, but in lieu of myronic acid, convertible as described into the volatile oil of mustard, it contains a non-volatile, bitter and acrid salt, termed *sulphocyanide of synapine* ($C_{17}H_{24}N_2SO_5$ or $C_{16}H_{23}NO_6CNHS$), *myrosin*, *gum*, *cellulose*, *and mineral matter*.
>
> Now it is on the volatile oil and the acrid and somewhat bitter salt that the pungency and acridity of mustard depends, and hence we see a strong reason why in the mustards of commerce the farina of the two species should be blended together; of the two active principles the volatile oil is by far the more important, and hence the seed of the brown mustard possesses the greatest commercial value. It should be stated that Henrie and Garot affirms that brown mustard contains the acrid principle as well as the white; this statement we have been able to verify as shown specially by the action of nitric acid, caustic potash, and ferric chloride on the alcoholic extract.

The acrid principle of white mustard appears to possess but little stability, and although it is stated by V. Babo to bear a temperature of 130°C., we find that it is readily affected by heat, and that it is not safe to evaporate the alcoholic solution containing it at a higher temperature than about 30°C. If subjected to a much higher temperature it quickly loses its acridity and acquires a bitter caramel-like taste.

Of neither brown nor white mustard had any percentage analysis been given until those made and published by ourselves in an article on mustard and its adulterations, in "Food, Water, and Air," for February, 1874; and in the few cases in which the quantities of any of the constituents are stated, they vary greatly according to different observers. Thus, according to Pereira, the *fixed oil* forms about 28 per cent. of the seeds of black mustard, while Watts puts the yield at 18 per cent. only, but white mustard seed, he says, furnishes 36 per cent. The *volatile oil* amounts to 0.20 per cent., according to Boutron and Robiquet; 0.55 per cent., according to Aschoff, and 0.50 per cent. according to Wittstock; all of which quantities are much below the mark, as will be seen hereafter. Now, as will be shown presently, there is little or no difference in the amount of fixed oil furnished by the two descriptions of mustard, that obtained by me from the farina of brown mustard reaching 35.701 per cent., and that from the white mustard 35.768 per cent. Again it is shown by the analyses given below that the volatile oil occurs in much larger quantities than those enumerated above, the amount which we have obtained from one sample being no less than 1.271 per cent.

Of both brown and white mustard we append the following original percentage analyses, first published in the article referred to:

BROWN MUSTARD FARINA.

	Per cent.
Water	4.845
Fixed oil	35.701
Myronic acid	4.840
Myrosin and albumen	29.536
Acrid salt	3.588
Cellulose	16.765
Ash	4.725
	100.000
Volatile oil	1.271
Nitrogen	5.068
Sulphur	1.413

The oil extracted by ether from the brown seed is of a bright and beautiful emerald green color, owing to the presence of the peculiar green principle described as one of its constituents. So deep and remarkable is the color of the oil that it would be easy, by means of a graduated scale of tints, to determine with very tolerable certainty the percentage of brown mustard contained in any samples of mixed mustard.

WHITE MUSTARD FARINA.

	Per cent.
Water	5.360
Fixed oil	35.768
Acrid salt	10.983
Myrosin and albumen	27.484
Cellulose	16.295
Ash	4.110
	100.000
Nitrogen	5.285
Sulphur	1.224

These analyses, whether regarded from a scientific or practical point of view, are possessed of much interest.

The small quantity of sugar found in mustard would, from the method of analysis pursued, be included under the bitter principle and the gum with cellulose.

Of the methods of analysis, Hassall writes:

Estimation of myronic acid.—Myronate of potash decomposes, under the influence of the nitrogenous matter contained in brown mustard, into volatile oil, glucose, and acid sulphate of potash. The quantity of each of these products of decomposition gives, therefore, by simple calculation, the quantity of myronic acid. One hundred parts of this acid yield 23.85 parts of volatile oil. From 40 to 50 grams of the mustard farina are placed in a flask of about one-half liter capacity; 250 c. c. of tepid water are poured over it, the flask closed with a cork, and the whole is well shaken. After twenty-four hours' standing the flask is connected with a Liebig's condenser, and its contents are heated to boiling. Into the receiver 30 c. c. of strong ammonia are poured, and the end of the condenser is dipped below the surface of the liquid. Water and the volatile oil pass over, the latter at first floating in the shape of oily drops on the surface of the liquid, which soon sink to the bottom, especially when the liquid is gently agitated. When the distillation is finished, which is the case when no more oil globules pass over, the receiver is closed with a cork and allowed to stand twenty-four hours. At the end of that time all the oil is dissolved and is now contained in the liquid in the form of thiosinnamin. This solution is evaporated on the water bath in a weighed platinum basin, the residue dried and weighed. The quantity of thiosinnamine obtained, minus one molecule of ammonia, represents the amount of volatile oil.

Estimation of the myrosin or albumen and of the sulphocyanide of sinapin.—The total amounts of nitrogen and sulphur contained in the mustard are next ascertained. The former by combustion with soda-lime in the well-known manner, the latter by deflagration of the mustard and oxidation of its sulphur in a mixture of nitrate of soda and carbonate of potash. The fused mass is dissolved in water or dilute acid, and the sulphuric acid contained in the solution is estimated by means of chloride of barium. From these data the amounts of the myrosin and of the sulphocyanide of sinapin, the acrid principle, are thus calculated; as much sulphur and nitrogen are first deducted from the totals of these substances obtained as is contained in the quantity of myronic acid previously determined. Next, the whole of the remaining sulphur and as much of the nitrogen as is required are then calculated into the acrid principle; lastly, the surplus nitrogen is calculated into myrosin, which has the same formula as vegetable albumen. But now, having got approximately the amounts of the acrid principle and of the myrosin, a further calculation has to be made, since myrosin contains about 1 per cent. of sulphur. This has to be deducted from the total acrid principle, a corresponding quantity of nitrogen being in its turn calculated into myrosin. By those acquainted with algebra it will readily be perceived that a more precise calculation may be made, but the results would not, even then, differ to any practical extent.

Acting on this method, Hassall made several analyses of genuine mustards of the trade and also of adulterated articles, which are here presented, merely dropping the third place in decimals, which is of no value.

Analyses of genuine mustard.

[Hassall, pp. 514–516.]

	Genuine mustard.	Genuine double superfine.	Genuine superfine.	Genuine Fine.	Pure.	Household.
Water	5.70	5.16	5.59	5.68	5.08	5.29
Fixed oil	36.49	35.94	34.71	35.24	33.98	36.75
Myronic acid	2.70	2.21	1.97	.92	.96	1.72
Myrosin and albumen	31.69	27.36	31.02	27.90	27.62	8.75
Acrid salt and bitter principle	5.72	9.09	7.10	10.06	11.26	27.48
Cellulose	13.37	15.58	15.29	15 55	16.81	3.69
Ash	4.33	4.66	4.32	4.65	4.29	16.32
	100.00	100.00	100.00	100.00	100.00	100.00
Oil of mustard	.71	.58	.52	.24	.25	.45
Nitrogen	5.34	5.05	5.46	5.16	5.21	5.03
Sulphur	1.31	1.42	1.25	1.30	1.40	1.31

Analyses of mixed and bulk mustard.

	Double superfine.	Fine.	Superior.	Suerls.	Alexander.	Lindsey.	Gilbert.	Goodman.	Clark.
Water	4.94	6.51	4.07	8.94	8.34	8.87	6.28	8.95	9.58
Fixed oil	27.52	23.16	25.17	23.88	29.60	21.54	22.06	26.90	18.31
Myronic acid	3.14	1.36	1.20	1.57	1.92	.98	1.13	1.82	.39
Acrid principle	1.85	5.81	4.31	6.45	3.15	6.21	4.25	5.18	7.03
Myrosin	23.16	19.50	23.24	14.48	13.89	21.76	15.30	15.58	20.82
Wheat flour and turmeric	22.99	27 20	25.82	33.81	30.52	25.21	38.82	30.56	32.81
Cellulose	13.05	12.84	11.50	7.08	8.99	11.69	8.41	7.27	8.65
Ash	3.35	3.62	3.79	3.79	3.59	3.74	3.75	3.74	2.41
Volatile oil	.85	.36	.32	.41	.50	.26	.30	.48	.10
Nitrogen	4.24	3.85	4.07	3.34	3.16	4.30	3.46	3.37	3.33
Sulphur	.95	.96	1.06	1.00	.90	.94	.82	.94	.91

"Of the first six analyses of genuine mustards" Hassall says that they "prove two things; first, that all the samples are genuine; this is shown by the quantities of fixed oil, nitrogen, and sulphur obtained; and that they consist of mixtures of the two mustards in different proportions, the higher qualities containing larger proportions of the brown mustard; that this is so is demonstrated by the different quantities of volatile oil obtained."

In the analyses of the adulterated mustards allowance in the calculation was made for the nitrogen of the wheat flour.

Hassall says:

> From an examination of the foregoing analyses it is apparent that genuine *brown* mustard should contain about 36 per cent. of fixed oil, at least 1 per cent. of volatile oil of mustard, about 4 per cent. of acrid principle, and that it should furnish about 1.5 per cent. of sulphur and 5 per cent. of nitrogen; that genuine *white* mustard should yield about the same amount of fixed oil, over 10 per cent. of acrid principle, and nearly the same amount of nitrogen and sulphur as the black; that the composition of genuine mustards, which are made up in various proportions of brown and white mustard seed, differs according to the quantities of each kind present, the relative proportions being determinable by analysis with considerable precision; that in the mixed or adulterated mustards the proportions of fixed and volatile oil, of nitrogen, and sulphur are all much reduced, according to the extent of the admixtures, these consisting in the mustards now reported upon in all cases of wheat flour and turmeric.

Thus the fixed oil was reduced in one of the samples from 36 per cent., the normal amount, to about one-half or 18 per cent., the volatile oil to 0.1 per cent., and the nitrogen to 3.32 per cent., while in another sample the sulphur was as low as 0.81 per cent. The amount of wheat flour and turmeric varied from 22.91 per cent. to 38.82 per cent., that is to say, from one-fourth to one-third of the article.

These results furnish an excellent basis for the examination of the mustards met with in our country. In addition, however, we have the work of several other investigators. C. H. Piesse and Lionel Stansell* have given their results of the analyses of several samples of pure farinas and their ashes, largely after the method of Hassall. Blyth copies them,† and adds several pages on the chemistry of mustard and its adulterations, adding nothing new to what has been quoted from Hassall, with the exception of a formula for calculating the percentage of added flour in mixtures from the amount of fixed oil found. He says:

Estimation of fat or oil.—This is particularly useful when wheat starch is the adulterating agent. Wheat flour does not contain more than 1.2 to 2.1 per cent. of oil; mustard, on the other hand, from 33.9 to 36.7 per cent. A weighed portion of the previously dried samples may be placed in an extraction apparatus, and from the oil found the following formulæ will serve as a guide to the amount of flour present:

$x =$ amount of mustard, $y =$ amount of oil found.

$$\frac{33.9\,x}{100} + \frac{1.2\,(100-x)}{100} = y \qquad \frac{36.7\,x}{100} + \frac{2\,(100-x)}{100} = y$$

according as the greater or less amount of oil is taken as being present in the pure farina and the flour.

Accepting the mean for mustard and 2.0 per cent. as the proper figure for flour, the formula would read more conveniently, it would seem,

$$x = \frac{y-2}{1.333}$$

where $x=100$ when the mustard is pure. Of the amount of ash Blyth says:

"The total ash of dried mustard averages 5 per cent. The highest number the writer has obtained is 5.3 per cent.; the lowest 5.088 per cent. Of this ash 1.2 at least is soluble in water; in other words, the ash of mustard consists of 30 parts per cent. soluble, 70 parts per cent. insoluble in water. It hence follows that if found above 5.5 per cent. mineral matters of foreign origin are present; if below 4 per cent. it is an indication of some organic adulterant."

Albert R. Leeds and Edgar Everhart‡ have taken up Hassall's and Blyth's work and shown that the latter's formula for calculating added flour from the percentage of oil found will not serve in all cases, as it is not uncommon to express some of the oil from the seed before grinding, to adulterate with oil cake or seeds, or to add cheap oils to the flour used as a diluent to cover any deficiency. They also analyzed a sample of pure farina of brown mustard according to a method devised by themselves as a modification of Hassall, in which the determinations should be direct instead of calculated. Briefly, it is as follows:

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* Analyst, **5**, 161–165, 1880.

† Foods and their adulterations, 485–486.

‡ Zeit. anal. Chem. **21**, 389–394, 1882.

METHOD OF LEEDS AND EVERHART.

Moisture and ash are determined as usual; oil, in a portion of mustard dried at 105°, by ether in an extraction apparatus, with subsequent drying at 100°. From the dried residue the *sulphocyanide of sinapin* and the *myronate of potassium* are extracted in a similar way by 50 per cent. alcohol. The extract is dried, weighed, and ignited, and from the sulphate of potash in the ash the myronate is reckoned and the sulphocyanide obtained by difference. The residue, containing *myrosin* and *cellulose* and a little coloring matter, is freed from alcohol and treated with one-half per cent. sodic hydrate solution. The washed residue is weighed and ignited for cellulose. The filtrate containing all the myrosin is nearly neutralized with dilute hydrochloric acid, 50 c. c. of Ritthausen's copper sulphate solution added, and then dilute sodic hydrate to near neutrality. The heavy green myrosin copper compound is filtered off, dried at 110° C., weighed, and ignited. The difference is the myrosin.

Analyses carried out after this method in triplicate are as follows:

	Per cent.	Per cent.	Per cent.
Water	6.78	6.90	6.82
Myronate of potash	.61	.61	.72
Sulpho-cyanide of sinapine	10.97	11.19	11.21
Myrosin	28.45	28.70	28.30
Oil	29.22	29.21	29.19
Cellulose (by difference)	20.24	19.55	20.06
Ash	3.73	3.84	3.70

The amounts of nitrogen and sulphur in this mustard were:

Nitrogen	5.337
Sulphur	1.489

Calculated from these figures according to the method of Hassall would be found the following:

Myronate of potassium	.61
Sulphocyanide of sinapine	10.71
Myrosin	28.52

Showing a close agreement with the direct determinations.

Direct methods are, however, usually preferable and this would, no doubt, be a good one were it not that dilute alcohol in the case of admixture of flour would dissolve so much of the albuminoid matter and ash of the latter as to invalidate the determination of myronate. It is therefore open to criticism.

E. Waller and E. W. Martin * in 1882 made an examination of mustards manufactured and sold in New York City, attention being paid to moisture, oil, and soluble and insoluble ash. They also examined mustard pastes, and compared their results with some pure mustards from the English market. Their analyses are as follows:

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* Analyst **9**, 166-170.

Dry mustard manufactured and sold in New York City.

No.	Moisture.	Fixed oil.	Soluble ash.	Insoluble ash.	Total.	Coloring.	Remarks.
197	6.15	21.17	.30	5.54	5.84	Martin's yellow..	Contains starch.
204	8.03	12.79	1.39	5.39	6.78	Turmeric..........	Contains starch and Ca SO_4.
206	7.35	12.54	.23	4.69	4.92	......do............	Ash fixed starch.
207	8.23	8.42	.15	1.90	2.05	Martin's yellow...	Contains starch.
208	8.50	10.92	2.90	13.15	16.05	Turmeric..........	Contains starch, Ca SO_4.
209	7.24	6.81	.10	3.55	3.65	......do............	Contains starch
213	7.65	13.32	.64	5.17	5.81	Martin's yellow...	Contains starch, ash-fused.
214	7.60	7.74	1.53	1.69	3.22	Turmeric..........	Contains starch.
215	7.15	9.02	.20	2.91	3.11	......do............	Contains starch.
216	5.45	20.57	.15	5.12	5.27	......do............	Contains starch.
217	6.50	8.59	1.52	5.65	8.17	......do............	Contains starch, Ca SO_4.
218	8.45	14.59	2.15	6.65	8.80	......do............	Contains starch, Ca SO_4.
219	6.62	22.56	1.62	4.86	6.48	....	No starch.
294	9.86	6.21	1.16	3.54	4.70	Martin's yellow...	

Mustard paste, German mustard manufactured in New York City.

No.	Moisture.	Acetic acid.	Oil.	Other organic substance.	Soluble ash.	Insoluble ash.	Total.	Common salt.	Oil on dry substance.	Metallic copper.
221	77.02	2.76	2.55	14.18	2.51	0.93	3.49	2.11	24.98	.001
222	81.52	1.98	3.50	10.67	1.77	.56	2.33	1.63	21.24	Trace.
237	79.62	2.43	3.90	12.60	2.52	.97	3.49	....	19.51	.009
242	76.54	3.69	4.57	11.53	2.69	.98	3.67	1.86	23.14	.003
244	81.45	2.94	3.73	9.09	2.14	.65	2.79	1.77	22.44	Trace.

Mustard flour (bolted) purporting to be pure.

No.	Moisture.	Oil.	Soluble ash.	Insoluble ash.	Total.	Remarks.
201	6.10	26.42	.21	5.92	6.21	New York manufacture.
220	5.50	25.70	.86	4.80	5.66	New York manufacture, Trieste and Bombay seed, mixed.
273	4.85	36.07	.175	3.725	3.900	English samples, whole seed.
274	4.75	41.70	.125	4.425	4.550	English samples, brown seed, ash-fused.

Ground mustard seeds.

No.	Kind of seed.	Moisture.	Oil.	Soluble ash.	Insoluble ash.	Total.
	American market:					
231	Bombay....................	7.52	36.96	1.25	4.37	5.62
232	Trieste....................	6.35	36.45	.70	3.70	4.40
233	California yellow........	4.95	34.00	.50	4.40	4.90
234	English yellow...........	6.10	35.46	.25	4.55	4.80
	English market:					
271	White.....................	7.10	34.45	.70	3.90	4.60
272	Brown.....................	7.30	34.71	.85	3.90	4.75

As comment they say:

The results obtained for oil on Nos. 201 and 220 led to inquiry, the result of which was the discovery that it is the regular practice of the mustard manufacturers here to express a portion of the oil from the ground mustard seed before working it up into the condiment sold as mustard. In these samples, as well as in No. 219, which was sold under guarantee of being pure mustard without admixture, no starch, coloring material, or other material known to be foreign to the mustard seed was found.

If we calculate, then, that these mustards have been made up from mustard flour similar to 201 and 220, and containing 25 per cent. of oil, by multiplying the percentages of oil given in the table by four, we shall get approximately the proportions of mustard flour present, in percentages.

These investigators also examined a specimen of English mustard which was mixed with starch, and yet contained a normal amount of oil which, on extraction, was decidedly more fluid than mustard oil. This points to the truth of the assertion that the extraction of the mustard oil is often covered by the addition of oil of inferior character.

The common use of Martin's yellow (Dinitronapthol) as a coloring matter is startling, as it can hardly be anything but injurious. They detected it by extracting the flour with cold, strong alcohol, evaporating, taking up with water and dyeing wool with it. Crystals, however, could not be obtained, but the authors came into possession of a sample of the coloring matter which was analyzed.

In Canada, large numbers of samples of mustard have been examined with results which have already been quoted, showing that the manufacture of mustard from mustard cake and the addition of farinaceous matter is as common there as elsewhere. In the report for 1885, out of fifty-one specimens, only 10 contained over 25 per cent. of fixed oil and no starch, and but three contained over 30 per cent. oil, as they should, if of best quality. The chief analyst, however, is of the opinion that the removal of the oil is an advantage, as the flour will keep better and be as pungent without it.

With the information of the nature which has been given a number of samples of mustard purchased in the open market in Washington, some of low grade obtained direct from Baltimore spice mills, and others in the whole seed from the importers and dealers, have been examined microscopically and chemically. They may be described as follows:

Serial No.	Price ½ pound (retail).	Remarks.
	Cents.	
4510	....	White seed.
11	30	Guaranteed pure.
22	20	Ground in District of Columbia.
27	20	Ground in Baltimore.
32	30	English brand.
36	36	Ground in Baltimore.
42	30	English brand.
50	20	
51	20	Ground in New York.
4241	....	Mohawk, N. Y.
71	....	Extra American, Baltimore, Md.
72	....	London, strong, extra, Baltimore, Md.
4971	30	Ground in Baltimore; extensively advertised as pure.
4835	....	White-seed flour, ground in laboratory.
86	....	White-seed husk, ground in laboratory.
99	....	California yellow mustard seed.
4900	....	California brown mustard seed.
1	....	English yellow mustard seed.
2	....	Trieste brown mustard seed.

The analytical determinations gave the following results:

Analyses of mustard.

[Whole-seed flour.]

Serial No.	Source.	Weight of 100 seeds, in grams.	Quality.	Water.	Ash.	Volatile oil.	Fixed oil.	Starch.	Crude fiber.	Albuminoids.	Undetermined.	Total.	Nitrogen.
4510	White seed	.635		5.57	4.29	.97	33.56	.00	5.40	28.88	21.33	100	4.62
4885	White flour			3.33	5.23	1.84	34.83	.00	9.05	25.56	20.16	100	4.09
4886	Seed husk	.480		6.17	4.99	.55	28.12	.00	9.50	23.44	27.23	100	3.75
4899	California yellow	.460		4.83	5.96	1.27	31.96	.00	8.50	31.13	16.35	100	4.98
4900	California brown	.135		4.11	4.88	1.35	36.63	.00	16.18	24.69	12.16	100	3.95
4901	English yellow	.419		3.11	4.07	2.06	31.51	.00	6.90	30.25	22.10	100	4.84
4902	Trieste brown	.425		4.62	5.61	.63	39.55	.00	10.84	25.88	18.87	100	4.14

[Commercial mustard flour.]

Serial No.	Source.	Weight of 100 seeds, in grams.	Quality.	Water.	Ash.	Volatile oil.	Fixed oil.	Starch.	Crude fiber.	Albuminoids.	Undetermined.	Total.	Nitrogen.
4511	(?)		Colored	5.97	6.55	.43	**18.16**	.00	7.35	37.44	24.10	100	5.95
4522	District of Columbia		Adulterated	9.38	**7**	.24	**11.20**	**21.15**	**2.95**	**20.63**	27.04	100	3.30
4527	Baltimore		do	9.73	4.40	.80	**9.85**	**29.14**	**2.40**	**13.31**	30.37	100	2.13
4532	English		do	5.84	4.90	2.11	30.84	**7.26**	4.63	25.88	18.54	100	4.14
4536	Baltimore		do	6.60	**9.70**	.50	**14.72**	**9.00**	**3.75**	**14.50**	41.23	100	3.92
4542	English		do	3 25	**3.65**	2.01	32.26	**8.50**	14.98	25.19	10.16	100	4.03
4550	(?)		do	5.80	**3.15**	.37	**7.89**	**40.50**	**2.43**	**14.38**	25.46	100	2.30
4551	New York		do	6.70	**1.90**	1.31	**6.50**	**45.00**	**2.97**	**13.63**	21.99	100	2.18
4871	Baltimore		do	4.57	**3.24**	2.02	**6.77**	**31.50**	**2.90**	**20.31**	28.60	100	3.25
4872	Baltimore		do	7.25	**3.35**	2.32	**5.54**	**34.63**	**1.23**	**19.63**	26.05	100	3.14
4971	Baltimore		do	6.03	5.98	.65	**19.46**	**5.70**	**3.48**	33.06	25.64	100	5.29

Abnormal figures in full-faced type.

DISCUSSION OF THE ANALYSES.

The results obtained with pure seed, ground in the laboratory, show that flour of mustard is fairly constant in its composition.

Water is present in small amount, as is generally the case in oil seeds, varying between 3 and 7 per cent. Hassall found from 5.16 to 5.70, and Waller and Martin 7.52 to 4.95.

Ash is quite constant between 4 and 6 per cent., so that the presence of foreign mineral matter is readily detected. Blyth places the variation between 5.1 and 5.3, Waller and Martin between 4.40 and 5.62, while in ground samples it falls as low as 2.05, Waller and Martin, or 1.90, our own results, owing to the addition of organic adulterants, such as wheat flour, or rises to 16 per cent. from the addition of gypsum. The determination is therefore an extremely valuable one.

Volatile oil is present naturally in the seed in but small amount. The percentage we have found to be rather variable, as much as 2.06 having been found in an English yellow seed and as little as .55 in another. Hassall found from .71 to 24 per cent. Its presence is not of importance.

Fixed oil is one of the most prominent constituents of the seed, varying in amount from 31 to 37 per cent. Waller and Martin give results varying from 34 to 37, and Hassall 34.71 to 36.49. It has become an almost universal practice, however, to express a portion of the oil, so that in good flour as little as 18 per cent. has been found.

Starch is entirely absent, a contrast to the cereal grains and many other seeds. Its addition is of course common.

Crude fiber varies in a way dependent on the method of milling and of determining its amount. Our samples were found to contain much more than the best flour of commerce, as our means of separating the husk are imperfect. With careful milling not more than 6 to 7 per cent. should be present, according to modern chemical methods. Hassall, on the other hand, found from 13 to 17 per cent.

Albuminoids make up a large part of the seed, varying from 25 to 32 per cent. Anything below 20 per cent. points to dilution with material poor in nitrogen.

The *undetermined* matter consists of gum and some unidentified substances soluble in alcohol, whose estimation is of no particular value as a means of detecting adulteration.

Our results, as a whole, agree closely with those of other investigators, so that for general reference the following standard may be used:

	Per. cent.
Water	3 to 7
Ash	4 6
Volatile oil	½ 2
Fixed oil:	
When from entire seed	31 37
When from cake	16 18
Starch	None.
Crode fiber	5 18
Albuminoids	25 32

When, however, the flour is ground from cake all the percentages may be somewhat increased.

As compared with the pure mustards the flours of commerce give results which show at once the universal extent of the adulteration which takes place, aside from any microscopic examination.

4511, purchased in Washington but ground elsewhere, has been deprived of nearly half of its fixed oil, that is to say, is ground from mustard cake. It is the only specimen which contains no farinaceous material, and is higher in albuminoids than any of the pure seeds or flours. This is due to the fact that abstraction of the oil raises the relative percentage of albuminoids, and this is not reduced at all by the addition of any diluent. The amount of fiber is relatively high for the same reason. It contains also a very small amount of mineral adulteration. The microscope shows the presence of turmeric as coloring matter.

This brand is perhaps the purest met with, as its only defects are lack of, oil which has been considered not a loss, the small amount mineral matter, and the presence of a little coloring matter.

4522, represented by two different specimens, has, in one case, that analyzed, been deprived of more oil than the preceding, has a considerable amount of gypsum in its ash, and 21 per cent. of starch in the shape of wheat flour. The relative percentages of fiber and albuminoids are thereby reduced somewhat, since 21 per cent. of starch would correspond to about 30 per cent. of flour. Color is given by turmeric. In the other case the changes were similar, except that no turmeric was added, but large amounts of white mustard hulls.

4527, ground in Baltimore, consists to a large extent of flour, between 40 and 50 per cent., and is made from mustard cake, the oil having been removed. The color has been restored by turmeric. The sample leaves on sieving quite a large amount of husky and fibrous matter contaminated with wheat flour and turmeric. The husks are not those of the original mustard, and have not been identified, but seem to resemble the exterior coats of ginger, and may represent an addition of spent ginger or ginger tailing.

4522, sieving leaves white mustard hulls and yellow corn meal, but no color; wheat flour is also present.

4551. This sample contains turmeric, wheat flour, and salt.

4532. An English brand, is made from whole seed and is diluted with but little flour and color, about 10 per cent. of the former.

4536 is made from mustard cake, contains a large amount of gypsum and some starch, and is colored (with turmeric). The large amount of undetermined matter would point also to the presence of other adulterants not identified.

4542 resembles the other English brand examined, 4532 being only altered by the addition of a little flour and color.

4550 and 4551 are perhaps the worst samples we have examined. They are made from cake, contain no mineral adulterants, but are more than half flour, and are of course much colored with turmeric. They were purchased from the same grocery. One also contained salt, detected by sifting.

4871 and 4872 are similar and but little superior. They were obtained directly from a Baltimore mill in a lot of spices, which were all adulterated.

4971 has been much advertised as quite pure, but was found to contain sand and flour, but no color. It is made, as usual, from seed cake. From these samples we learn the quality of the ordinary flour mustard of the groceries. It is not good, and certainly demands reform.

PEPPER.

The ordinary black and white peppers of commerce are the fruit of the true pepper plant, *Piper nigrum*, which grows in the East and West Indies. Red pepper, or cayenne, is not a pepper, but the fruit of several species of *Capsicum*. Under the title of "pepper," therefore, attention will be confined to the genus *Piper*, reserving for a separate chapter the remaining substances which are commonly miscalled peppers.

The pepper plant is a perennial climbing shrub, with a small, round, sessile, fleshy fruit, which grows spontaneously on the Malabar coast, and whose culture has been extended to Siam, Hindostan, Indo-China, Malacca, Singapore, Penang, Ceylon, Sumatra, Java, Borneo, and the neighboring islands, and to a small extent in Guiana and Cayenne.

The greatest production is in Sumatra, and the ports of export are principally Singapore and Penang, the Malabar pepper coming from Tellicherry. Our imports are principally through England, and not direct, and it seems that, in England at least, it is customary to mix peppers of different origin in grinding, taking Malabar for weight, Penang for strength, and Sumatra for color.

Of the other characteristics and history of pepper a most complete account may be found in "Flückiger and Hanbury's Pharmacographia."* Of its preparation for the market these authors say:

> When one or two berries at the base of the spike begin to turn red the whole spike is pinched off. Next day the berries are rubbed off with the hands and picked clean, then dried for three days on mats or on smooth, hard ground or on bamboo baskets near a gentle fire.

As thus prepared it is the black pepper of the trade.

When the berries are allowed to ripen, and the black outer pericarp is removed on drying, they are known as white pepper.

> The grains of white pepper are of rather larger size than those of black, and of a warm, grayish tint. They are nearly spherical or a little flattened. At the base the skin of the fruit is thickened into a blunt prominence, whence about twelve light stripes run meridian-like toward the depressed summit. If the skin is scraped off the dark-brown testa is seen inclosing the hard, translucent albumen. In anatomical structure, as well as in taste and smell, white pepper agrees with black, which, in fact, it represents in a rather more fully-grown state.

A study of the structure of black pepper will, therefore, furnish every information in regard to the white. There are also two species of *Piper* which furnish a berry used in a similar way to that of *Piper nigrum*. They are known as long pepper, *Piper longum* and *Piper officinarum*. Their structure is similar to that of the common pepper, with some characteristic differences. It is difficult to say how far they are an article of commerce in this country. They come principally from Penang and Singapore, being brought from Java and other places.

The structure of black pepper is described as follows in the work last quoted:

> The small, round berry-like fruits grow somewhat loosely to the number of twenty to thirty on a common pendulous fruit stalk. They are at first green, then become red, and, if allowed to ripen, yellow, but they are gathered before complete maturity, and by drying in that state turn blackish gray or brown. If left until quite ripe they lose some of their pungency and gradually fall off.
>
> The berries after drying are spherical, about one-fifth inch in diameter, wrinkled on the surface, indistinctly pointed below by the remains of a very short pedicel, and

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* London, MoMillan & Co., 1879.

crowned still more indistinctly by the three or four lobed stigma. The thin pericarp tightly encloses a single seed, the embryo of which, in consequence of premature gathering, is undeveloped and merely replaced by a cavity situated below the apex. The seed itself contains within the thin red-brown testa a shining albumen, gray and horny without and mealy within. The pungent taste and peculiar smell of pepper are familiar to all.

The transverse section of the grain of black pepper exhibits a soft yellowish epidermis, covering the outer pericarp. This is formed of a closely-packed yellow layer of large, mostly radially arranged, thick-walled cells, each containing in its small cavity a mass of dark brown resin. The middle layer of the pericarp consists of soft tangentially extended parenchyme, containing an abundance of extremely small starch granules and drops of oil. The shrinking of this loose middle layer is the chief cause of the deep wrinkles on the surface of the berry. The next inner layer of the pericarp exhibits towards its circumference tangentially arranged, soft parenchyme, the cells of which possess either spiral striation or spiral fibers, but towards the interior loose parenchyme free from starch and containing very large oil cells. The testa is formed in the first place of a row of small yellow thick-walled cells. Next to them follows the true testa, as a dense, dark brown layer of lignified cells, the individual outlines of which are indistinguishable. The albumen of the seeds consists of angular, radially arranged, large-celled parenchyme. Most of its cells are colorless and loaded with starch, others contain a soft yellow amorphous mass. If thin slices are kept under glycerine for some time, these masses are slowly transformed into needle-shaped crystals of piperin.

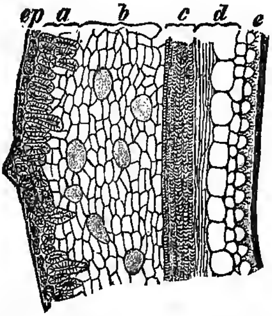

FIG. 11. Pepper husk, cross-section. *ep*, epidermis; *a*, stone cells; *b*, parenchyme with oil cells; *c*, spiral vessels; *d*, inner parenchyme; *e*, inner layer of stone cells. (After Schimper.)

Of the structure described so well in the preceding lines, of a portion of which a diagramatic illustration is given in Fig. 11, after Schimper, only parts are readily found in the powdered pepper of the shops. The angular cells of the interior of the seed are of course the most prominent, and when once seen their characteristic form and contents are easily recognized again. The structure of the outer coats is made out with more difficulty. It is well, before attempting to do so on a ground pepper, to soften some whole black and white pepper-corns in glycerine and cut sections from various parts of the exterior of the berry. Taking the white pepper first it will not be found difficult in such sections mounted in glycerine to pick out three layers of different cells composing the outer coat of the corn, beside the angular large cells of the interior, which are filled with starch and piperine, the latter being yellow in color. The first of these layers and outer one is made up of colorless large loosely-arranged cells, with some fibers, more compact toward the exterior than the interior of the layer and carrying globules of oil. This layer makes up the principal part of the husk of white pepper. The second layer is a part of what Flückiger calls the testa, and consists of small yellow cells, thick walled and closely appressed. Next the third layer and second portion of the testa consists of liginified brown cells, which in their transverse appearance resemble some of the cells of mustard hulls, the individuality not being made out easily owing to the thickness of the walls. Having become thoroughly familiar with these appearances the white ground pepper should be examined and will be found to differ in the way in which these coats are presented. They can be recognized, however, and must be studied until thoroughly understood. The presence of the least portion of adulterant is then readily detected.

The black pepper is not as simple in its arrangement as the white. The maturity of the latter gives its structure more distinctness, while in the black the more or less shrunken character of the berry renders the recognition of the various tissues difficult. In a section from the exterior of a softened black pepper the interior coats, after what has been learned with the white, will be quickly recognized, but will be found to not be as plainly developed. The coats of the outer pericarp, which in the white pepper were wanting, will be found to be dark-colored, shrunken, and confused, so that it will require much study to discover the forms of cell which Flückiger describes. It will be found easier, perhaps, in the ground black pepper. There the structures already recognized in the ground white pepper will be seen and in addition dark-brown particles, portions of the outer coats. Careful examination of different particles will detect some which consist of the elongated vertical exterior cells, containing resin, while others are the shrunken parenchyma cells of the second layer, whose structure is indistinct.

Flückiger calls the first layer yellow, which hardly seems correct, as the appearance is nearly black. It is unnecessary, however, to attempt a minute study of these cells, as one is only required to be able to recognize their appearance and in addition to know something of the relative proportion of ground pepper which they should form as they are added in excess as pepper dust, the waste hulls of pepper being often used as an adulterant. The colored portion of a ground black pepper it will be found divides itself into two classes, the dark particles which have just been mentioned and the deep reddish ones, which are made up of the testa of the seed and its adherent parenchyma. The two will be readily recognized and distinguished from adulterants by the investigator.

The differences in the appearance of the peppers from various sources is sufficiently marked to be readily noticed when samples of each are placed side by side, but otherwise it is almost impossible to identify them. The best method of judging their quality and the one in use in the trade is by weight. Malabar is considered the heaviest. Blyth gives the following figures:

100 peppercorns of—	Grams.
Penang	6.2496
Malabar	6.0536
Sumatra	5.1476
Trevy	4.5736
Tellicherry	4.5076

Tellicherry is the Malabar or West Coast port, so that variations must occur, as would be natural, in different samples.

We have found some samples to weigh—

Serial No.	Source.	Weight per 100 grams.	Percentage of dust and dirt.
	Black.		
4514	Unknown	5.9000	
4840	Unknown	5.4600	
4894	Acheen	4.525	2.5
4895	West Coast	5.085	4.3
4896	Singapore	4.870	8.3
	White.		
4516	Unknown	5.1300	
4898	Singapore	4.9600	1.4

The white pepper is of course the cleaner.

ADULTERANTS.

The common adulterants to be found in peppers are said to be flours or starches of cereals and potatoes, sago, mustard husk, linseed and capsicum, pepper dust, sawdust, gypsum, and other odds and ends.

Of the adulterations of pepper Blyth enumerates many, among them the celebrated pepper dusts designated as “P. D.”, “H. P. D.,” and “W. P. D.,” and known as pepper dust, composed of linseed cake; hot-pepper dust, composed of mustard husks chiefly, and white-pepper dust, composed of ground rice. In this country the use of ground corn or rice, mustard hulls, cocoanut shells, and other similar refuse is very common, but whether they have been derived from goods sold as H. P. D. and W. P. D.” we were unable to ascertain. Other refuse is also frequently found in cheap peppers, but sand does not seem to be as often added here as abroad. We have found the white peppers much freer from adulteration than the black. It is learned from Dr. Ellis, of Toronto, that roasted cocoanut shells are in common use now as an adulterant of all spices, and are of course easily introduced into peppers. They have not been found in the specimens which we have examined. Pepper husks, mustard hulls, yellow corn, cracker dust, charcoal, and mineral matter have been detected by their characteristic appearances. The presence of pepper husks and charcoal is generally known by the immensely increased proportion of black particles in the field, as appears in Fig. 43, Plate XVII. A careful sorting of the coarser particles under a low power of the dissecting microscope and selection, for example, of the shining white grains of rice or yellow particles of corn from the more oily pepper cells, enables us to examine the starches separately with plain and polarized light. The appearance of the true pepper powder and one in which rice starch is present is given in Plate XXII, Figs. 43 and 44. Pepper starch is so much smaller than any other which we meet with, that it is not easily confused with it. Fig. 65, Plate XXVIII, gives an idea of its size as compared with others. With the dissecting microscope it is also convenient to go over the pepper powder and pick out any other class of suspicious particles for examination with higher power. With a little practice it soon becomes easy to tell an adulterated specimen even with a cursory examination with a lower power or a hand lens. In this way, in our experience, one or two foreign substances have been found which have not been identified, but which were evidently parts of the husks or coverings of some seed or fruit. The shell of the cocoanut, as has been said, has not been met with as an adulterant, but it is not at all difficult to identify, as its structure, with its innumerable stone cells and fibrous tissue, is very characteristic after treatment with Schulze's reagent.

The microscopic examination thus gives very certain indications of the quality of peppers, although frequently the eye without aid will detect a fictitious appearance. The chemical examination, on the other hand, as will be shown, serves as a reliable means of confirmation in many instances, and gives evidence of the quantity as well as kind of the adulteration. A considerable amount of information has been accumulated in regard to the proximate composition of peppers. Flückiger quotes authorities for the statement that black pepper contains from 1.6 to 2.2 per cent. of volatile oil, of the character of a terpene and optically active; also a nitrogenous substance known as piperine to the extent of 2 to 8 per cent. Husemann and Hilger* give reference to papers by several investigators (see biobligraphy), and describes piperine as soluble in alcohol, and less so in ether. This substance, which is characteristic, will be found, therefore, in the ether extract of the spice, together with the volatile oil.

Blyth has made an examination of several peppers, and gives results which are useful for reference.

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* Die Pflanzenstoffe, B. 2, S. 486.

Analysis of ash of Tellicherry pepper.

	Per cent.
Potash	24.38
Soda	3.23
Magnesia	13.00
Lime	11.60
Iron	.30
Phosphoric acid	8.47
Sulphuric acid	9.61
Chlorine	7.57
Carbonic acid	14.00
Sand	6.53

The sand, he finds, the most variable constituent, but never above 9 per cent. of the ash except in cases of willful adulteration. Phosphoric acid averages 8.5 per cent. of the ash, which is considered characteristic. He has also identified nitrates in peppers, finding in—

	Per cent.
Penang	.04470
Malabar	.03858
Tellicherry	.08860
Sumatra	.06560
Trang	.11870

The average proximate composition he finds to be—

	Per cent.
Volatile oil	1.04
Acrid resin	1.77
Piperine	5.17
Substances soluble in water, gum, starch, and other matters substracting ash	14.74
Substances insoluble in alcohol and water	67.75
Water	9.53
	100.00

Following are some analyses made by Blyth in 1876:

	Hygroscopic moisture.	Piperine in pepper, dried at 100°.	Resin in pepper, dried at 100°.	Aqueous extract in pepper, dried at 100°.	Ash in pepper, dried at 100°.	
					Soluble in water.	Total.
Penang	9.53	5.57	2.08	18.33	2.21	4.18
Tellicherry	12.90	4 68	1.70	16.50	3.38	5.77
Sumatra	10.10	4.70	1.74	17.59	2.62	4.31
Malabar	10.54	4.63	1.74	20.37	3.45	5.19
Trang	11.66	4.60	1.70	18.17	2.53	4.77
White pepper (com.)	10.30	5.60	2.05	......	.56	1.12
Long pepper	....	1.80	.80	16.82	4.47	8.30

The process for the estimation of piperine approved by Blyth is extraction with petroleum ether and treatment of the extract with sodic hydrate to remove the resin. Subsequent investigations by Lenz condemn this method, however.

More recently Rottger[*] has investigated the pepper-corn with especial consideration of the question of detection of adulteration, and decided that the examination of commercial peppers should be directed to determinations of the inorganic constituents, the percentage of water, and the microscope, with determinations of the soluble and insoluble ash and piperine in certain instances. He decides that the alcohol and ether extracts are of no value, quoting great variations in his results and those of others. He found—

	For ether extract, indirectly determined.	For alcohol extract of 90 per cent.
	Per cent.	*Per cent.*
White pepper	8.0 to 10.7	10.0 to 11.8
Black pepper	7.9 12.1	12.3 18.7

Borgman, Wolff, and Biechele[†] likewise have determined the amount of alcohol extract, but have neglected the ether extract. It seems difficult to understand why the alcohol extract should be selected, as the ether is much simpler, and refers almost directly to the amount of volatile oil and piperine which the spice contains, without so much of the indefinite resin and sugar which the alcohol extracts. The determination of water was made as follows by Rottger: The powdered pepper was placed for three hours over sulphuric acid, and a portion then weighed out and dried at 100° C. for one and a half hours, then for a quarter of an hour at a time until it begins to gain weight. He found the variations to be between 12.6 and 14.7 for black pepper, and 12.9 and and 14.5 for white pepper.

This striking agreement, he considers, makes this determination of value. It is open to serious criticism and is of little value, in our view, for the following reason: By consulting our investigations on the hygroscopic character of organic matter in a state of fine division,[‡] it will be seen that allowing peppers to remain over sulphuric acid for three hours would not accomplish the end desired, at least in our climate, viz, reducing the moisture in all to a constant figure, and in addition Rottger takes no account of the volatile oil lost at 100°. In our analyses we find, in fact, much less water than he does, as is usual in all organic material in our drier climate, and no greater variation among the peppers than the adulterated specimens. Practical tests of the method also proved unsatisfactory.

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[*] Ber. uber d. 4 Ver. Bayrisch. Vertreter der angew Chemie, 97–102.

[†] Vide Bibliography.

[‡] Bull. No. 4, Div. of Chem., Department of Agriculture.

The per cent. of ash Rottger finds for black pepper to be between 3.4 and 5.1 per cent., white pepper between .8 and 2.9 per cent., with an exception in the case of Lampong pepper, which has 6.4 per cent. He concludes, with other investigators, that 6 is the highest allowable figure for ash in black pepper, with anything above 5 as suspicious. Three per cent. is, in the same way, the highest allowable figure for white pepper..

Of the composition of the ash he says:

> The facts point to the conclusion that an exhaustive investigation of the mineral constituents in many cases may be of benefit in forming an opinion of the quality and purity of peppers. *

He found the following extremes:

	Black.	White.
Fe_2O_3	2.2	
Mn_2O_3	.89	
K_2O	27.4 to 34.7	5.1 to 7.1
Cl	5.6 8.7	.5 .9
SiO_2	1.5 6.3	1.0 2.6
P_2O_5, water sol	.11 .91	....
P_2O_5, insol	8.2 12.5	10.8 30.7

He gives the following detailed analyses:

Percentage composition of the pepper ash.

	Black pepper.		White peppor.	
	Unknown origin.	Malabar, 1883.	Unknown origin.	Singapore.
SiO_2	6.36 to 1.61	1.54	2.62	1.46
HCl	5.59 6.83	8.71	.58	.90
SO_3	4.03 4.05	4.00	3.24	3.75
CO_2	17.28 20.10	19.17	11.90	10.01
P_2O_5	11.10 9.46	11.06	29.34	30.75
K_2O	32.49 34.72	27.39	5.10	7.15
Na_2O	1.55 4.77	5.50	.74	.84
CaO	16.07 13.55	15.02	35.12	31.05
MgO	3.31 4.46	7.56	9.54	11.64
F_2O_3	2.16 .99	.85	2.22	1.86
Mn_2O_3	.81	.18	.89	.21

Rottger then discusses the determination of the piperine, and allows that for simplicity and accuracy drying the powder with milk of lime and extraction with ether is to be preferred. When the ether is pure, dry, and free from alcohol we find lime is of little use, or rather a complication, and that for purposes of detecting adulteration the ether extract alone furnishes all the information desired.

Lenz† has determined the amount of sugar produced by inverting the starch in fourteen peppers, and in the common adulterants, with acid. He found all the samples gave an equivalent of more than 50 per cent.

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* Halenke has more recently discussed this subject: vide bibliography, Appendix A.

† Zeit. Anal. Chem., 1884, **23**, 501.

of sugar on the ash free organic matter of the pepper, while all the adulterants gave less than 30 per cent., with the exception of those which are starchy, as flours and meals.

Rottger repeated this determination, showing that some other substances besides starch were inverted by the acid, and obtained the following results:

Black pepper sugar equivalent	57.2 to 60.3
White pepper sugar equivalent	59.6 to 74.4

The Lampong pepper gave only 41.70. Such great variations he considers fatal to the method as a means of detecting adulterations. His conclusions seem not entirely justified, as a review of Lenz's figures will show.

Lenz in his paper affirms that any extract determination is useless, as with various adulterants the results may be very close to actual pepper. Petroleum ether he shows to be unreliable, the amount of extract depending on the kind of extraction apparatus used. There certainly should be no difficulty of purely manipulative detail of this description, for if sufficient time is given the solvent will work the same under all conditions, but not less than twenty hours' continuous extraction should be allowed, and it is not fair to generalize on an analysis where the extraction was continued less than that time, the poor results being due only to a probable faultily manner of manipulation, as we have met with no such trouble.

Lenz's conclusions seem hardly just, and while there may be cases where adulterants would not be detected by an extract determination, in the majority it is a great assistance. Lenz also refers to the method of separation of the powdered particles of pepper adulterants by means of liquids of certain specific gravities, and pronounces it usuitable. He prefers the treatment with iodine solution and the selection of the particles not blued for examination under the microscope as adulterants. We have found it perfectly simple and desirable to use the method of separation with sieves, and to examine the coarse powder, with or without treatment, under the microscope, when, with a little experience, it is easy to distinguish and recognize the source of particles not pepper after a careful examination with high powers.

He also casts doubt upon the value of an ash determination, and owing to the great variations which he finds quoted as possible, recommends calculating all results on an ash free basis. He, however, admits that a carefully and properly cleaned pepper-corn will not usually contain more than 6 per cent., and his strictures, therefore, do not seem to be consistent. He places reliance only on the process of his own invention, determinations of sugar corresponding to the starch and other invertible substances, with supplementary determinations of water and ash and microscopic examination. His results were as follows:

List of equivalent percentage of reducing sugars in ash free organic substance of peppers and adulterants.

No.	Name.	Ash.	Dry residue.	Ash free dry residue.	Sugar equivalent in sample.	Sugar equivalent in ash free dry sample.	Remarks.
1	Black Batavia pepper	3.85	87.68	83.83	43.8	52.3	Inverted directly.
2	Long pepper (2)	8.68	88.77	80.12	44.2	55.2	Inverted after extraction with water.
3	Black Singapore	3.62	86.88	83.26	43.9	52.8	Inverted directly.
4	Do	3.62	86.88	83.26	45.0	54.1	Inverted after extraction with alcohol.
5	Do	3.62	86.88	83.26	44.1	53.0	After water.
6	White pepper	.99	87.59	86.60	51.8	59.9	Do.
7	Palm-cake meal (1)	3.71	89.76	86.05	22.7	26.4	After water and alcohol.
8	Do	3.71	89.76	86.05	19.1	22.2	After water.
9	Do	3.71	89.76	86.05	22.7	26.4	Direct.
10	Palm-cake meal (2)	3.65	89.35	85.70	22.4 22.5	26.1 26.2	Do.
11	Do	3.65	89.35	85.70	19.7	23.0	After water.
12	Palm-cake meal (3)	3.54	89.88	86.34	19.4	22.5	Do.
13	Palm-cake, pure	1.89	93.44	91.55	11.1	12.1	After ether and alcohol.
14	Pepper husks	15.61	89.50	73.89	11.5	15.6	After water.
15	Pepper husks picked out of commercial pepper.	9.21	88.64	79.43	13.0	16.4	Do.
16	Commercial pepper husks.	20.29	88.42	68.13	11.5	16.9	Do.
17	Commercial pepper powder containing palm-cake meal.	5.15	88.70	83.55	35.9	43.0	Do.
18	Pepper (3), with 28 per cent. of palm-cake meal (3).	3.59	87.88	84.29	36.0	42.7	Do.
19	Same with 42.4 per cent.	3.58	88.38	84.80	33.7	39.7	Do.
20	Walnut shells	1.04	89.34	88.30	17.7	20.0	Do.
21	Buckwheat meal	2.10	86.58	84.48	56.1	66.4	Direct.
22	Dried and roasted bread.	1.15	100.00	98.85	....	86.3	Do.
23	Do	1.15	100.00	98.85	....	62.6	After water.

Lenz's method of inverting was this: 3 to 4 grams of the substance to be examined was treated for three to four hours in a flask with 250 c.c. of water, being repeatedly shaken. It was then filtered off, washed with water, and the moist powder washed back into the flask, and to 200 c.c. of water 25 c.c. of 25 per cent. hydrochloric acid added. The flask was provided with a cork and tube one meter long and placed in a bath of boiling water and kept there three hours, with shaking. It is then made up on cooling to 500 c.c., after careful neutralization with soda. This liquid was then titrated with Fehling solution. When palm-cake was present it was found necessary to add a little zinc chloride to clear the solution. Most of the surrogates for pepper used in Germany Lenz found to be free from starch, so that this method would seem to be of wide applicability. He remarks, however, that other substances besides starch may be inverted, and for this reason it is necessary to adhere closely to one method of working. We shall from our own experience and that of others learn more in regard to the capabilities and usefulness of this method.

Haslinger and Moslinger* have also published some observations on pepper and its adulteration which are of interest. They show that in Germany the addition of grains of paradise, the seed of *Amomum Melegueta*, is a common practice whose detection can be accomplished most readily with the microscope, after a study of the structure of the grains of paradise, which is very characteristic. The starch cells are larger than in pepper, 3 to 6 times as long as hard, but the starch itself is scarcely distinguishable. The cells are arranged in parallel lines forming bundles pointed at the end. The cells remain white when treated with hydrochloric acid, while those of pepper are yellower.

These writers also looked into the ash of commercial samples of pepper and found from 4.1 to 27.0 per cent. of inorganic substance insoluble in hydrochloric acid where in peppers ground by themselves only .3 and .8 per cent. occurred. They also found pepper siftings, pepper husks, and other refuse in use as adulterants and followed up Lenz's suggestions as to determination of reducing sugar produced from the samples by acid, by examining pure pepper corns ground by themselves, and also pepper husks, and extending the determinations to cellulose also. The "dextrose," as they denominate it, was determined according to Allihn, the "cellulose" according to Henberger. The results were as follows:

	Dextrose.	Cellulose.
Black pepper, pure	56.0	15.6
Pepper husks, pure	16.4	45.0
Four samples for investigation:		
I	46.4	23.4
II	40.8	28.1
III	44.9	23.3
IV	41.5	26.8
Pepper siftings	21.6	37.4

From these figures they give the following formulæ for calculating adulterations where:

x=per cent. of pure pepper.
y=per cent. of hulls.
s=per cent. of dextrose or cellulose in sample.
a=per cent. of dextrose in pure pepper.
b=per cent. of dextrose in husks.

For the dextrose figures:

$$x=\frac{100s-1640}{39.6}$$

For the cellulose figures:

$$x=\frac{4500-100s}{29.35}$$

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* Ber. 4. Ver. Bay. Verterter ang. Chemie, 104–110.

Calculated on this basis the four samples were found to consist of:

	Pure pepper husks and pepper.	
	On the ground of dextrose det.	On ground of cellulose det.
I	75.8 and 24.2	73.5 and 26.5
II	61.6 38.4	37.5 42.5
III	72.1 27.9	74.0 26.0
IV	63.3 36.7	65.4 34.6

These figures show a fair agreement, and, in the opinion of the authors, entitle the method to consideration. They condemn, however, severely placing any reliance on determinations of the alcohol extract.

Weigmann* has also examined a number of pure peppers, and obtained results which do not harmonize with those of Lenz. He finds:

	White.	Black.
Ash	1.0 to 3.0	3.0 to 7.0
Starch (Lenz method)	55.0 63.0	32.0 44.0
Fiber	5.0 6.0	12.0 15.0
Water	12.0 to 15.0	

Our experience with the inversion of starch by acids is such as to make it seem very probable that, without the necessary attention to details, it would be very possible to obtain such results as those last given from lack of complete conversion of starch to reducing sugar or the inversion of other substances, and in the discussion of our analyses this will be referred to.

In the second report† of the Laboratoire Municipal de Paris the subject of pepper and its adulteration as practiced in France is discussed. There whole peppers are made artificially from plaster, gum, and a little pepper, and the ground article with the most diverse substances, such as hemp-seed cake, colza, rape and beechnut cakes, starches, residues from the manufacture of potato starch, mineral matter, sweeping of spice warehouses sold as pepper dust, and so forth. The residue from the manufacture of potato starch has, after fermentation, a pungent taste which makes it a desirable adulterant, but the most common one is the powdered kernel of the olive, which is yellowish white in color and possesses all the outward characteristics of white pepper and gives practically the same amount of ash. To give this powder the proper taste and pungency there was added Cayenne, powdered bay leaves, and dried and powdered orange skins. The mixture is recognized without difficulty by chemical and microscopic examination, the latter of which gives an absolute proof of the character of the substance.

It is recommended to separate the olive kernels from pepper by the use of a mixture of glycerine and water of a Sp. Gr. of 1.173 at 10° C., in which the former sink. After decantation they can be examined

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* Rep. anal. Chem. **6**, 399–40.

† Documents sur les falsifications des Matieres alimentaires, Paris, 1885, p. 688–695.

microscopically. The structure is that characteristic of the hard thickened schlerenchyma or stone cells, polyhedral and elongated in shape with thickened walls and little filiform canals. They are most readily made out with polarized light, toward which they are optically active, while the remaining particles of pepper or pepper husks are not seen in the dark field, being inactive.

Of this adulterant H. Rabourdin has made some investigations, publishing his results in an interesting paper,* giving a general account of the various peppers of commerce and the use of olive stones as an adulterant, as well as other materials already mentioned. He finds no difficulty in recognizing the falsification owing to the presence of the numerous sclerenchyma or stone cells, which are distinguished readily with polarized light, but the quantitative determination is more difficult. This, however, he affirms, can be accomplished by determining the residue left on boiling the suspected sample for one hour with 1 per cent. sulphuric acid after thorough washing with water. In the course of the operation the presence of the olive stones is apparent from their clinging in the form of a reddish powder to the walls of the flask or beaker in which the digestion has taken place, and separating more or less from the pepper hulls on account of their different specific gravity.

For pure peppers the following percentage of residue was found:

	Per cent.		Mean.
Tellicherry	30.3 to	27.8	29.4
Alepy	31.5	31.8	31.7
Saigon	28.7	29.2	29.0
Singapore	33.2	34.0	33.7
Penang	33.8	34.2	34.0
Sumatra		33.0	......
Java	37.5	37.7	37.6
White, pure		17.5	17.5
Powdered, pure	29.2	29.0	......
Do	30.5	35.5	......

On the other hand olive stones were found to contain:

	Per cent.		Mean.
Coarse gray	74.2 to	75.0	74.5
Fine gray	74.2	74.6	74.4
Coarse white		74.5	......
Fine white		74.5	......
Pepper dust		65.5	......

From these figures the following factors are derived:

	Per cent.
White pepper	17.5
Malabar, Tellicherry, and Saigon	30.0
Alepy	32.0
Others known as light peppers	35.0
Olive stones	74.5
Pepper refuse	75.5

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*J. de Pharm. et de Chimie, [5], 9, 289-297.

And if y is the per cent. of residue, a the per cent. in pure pepper, b the per cent. in olive stones, the amount of addition can be calculated from the following equations:

$$x+y=100 \qquad ax+by=p$$

$$y=\frac{p-a}{b-a}$$

where b can be replaced by 74.5 or 65.5 as the case may be.

Working in this way 10 ground peppers, guaranteed pure, were found to contain from 40 to 60 per cent. of olive stones, and these contained "P.D." to the extent of 14 to 20 per cent.

Control experiments with known mixtures seem to have been satisfactory.

The same author also extended his experiments to a comparison of the properties and separation of the cellulose derived from a mixture of pepper and olive stones, depending upon the difference in specific gravity, with results which can be found in his original paper.

The residues of starch factories always contain enough starch grains to make their identification easy.

Very recently Chas. Heisch* has given in the Analyst his experience in the examination of peppers, to see if there were any reliable mode of judging of the amount of adulterants in adulterated samples except by determinations of ash which give no indication of added organic matter. He also endeavored to find if it were possible to cleanse pepper corns so that the ash should be free from sand. Assisted by a large buyer and grinders of pepper he found that as the result of the grinder's experience no ground pepper should be sent to market which contains over 6 per cent. of total ash. In this respect they agree with the Bavarian chemists. He also placed some faith in the determination of starch and calculated, as did Lenz, his results on dry ash free organic matter.

The starch was estimated "by boiling the finely ground pepper for three hours with 10 per cent. hydrochloric acid, and taking the rotation in the resulting liquid." He endeavored to check his results by determining that there was insufficient "gum or other matters to affect the rotatory power" but neglects to see how much of the substances allied to cellulose are converted into optically active substances which would probably be important. Stating the results, however, as reducing sugar equivalent to pepper with Lenz, the error becomes of slight importance.

The determination of piperine was rough, and the results seemed of little service.

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*Analyst, **2**, 186-190, October, 1886.

The results were as follows:

No.	Name of sample.	Water.	Ash of dry pepper.					On ash and water free pepper.		
			Total.	Soluble in H_2O.	Soluble in HCl.	Insoluble.	Alkalinity as K_2O.	Starch.	Alcohol extract.	Piperine.
	Black Pepper:									
1	Acheen Penang	9.46	8.99	1.54	3.07	4.38	.72	48.53	12.26	6.04
2	Trang	9.22	8.85	1.60	3.83	3.42	.81	54.06	12.28	4.05
3	Singapore	14.36	5.41	2.07	2.52	.82	.91	56.24	12.41	7.14
4	Tellicherry	13.76	5.28	3.34	1.90	.04	1.41	56.67	12.67	6.88
5	Penang	12.98	6.45	3.10	2.44	.91	1.19	51.66	16.20	9.38
6	Tellicherry (brushed)	13.01	6.41	2.38	2.84	1.20	1.57	55.87	13.62	7.86
7	Gray light dusty Singapore	13.94	5.39	2.48	2.18	.73	1.09	54.93	11.62	6.30
8	Good B, Singapore	14.10	4.35	2.48	1.51	.36	1.14	54.54	10.47	6.66
	White Pepper:									
9	Penang	15.86	3.78	0.62	2.80	.36	.22	77.68	9.73	5.54
10	Singapore	17.32	1.28	.22	0.85	.22	.00	76.35	9.49	6.14
11	Siam	13.67	1.81	.25	.92	.69	.11	76.27	9.23	5.13
	As ground for market:									
12	Fine white	13.90	1.58	.16	.90	.52	.00	75.31	10.60	4.51
13	Finest	14.13	2.18	.50	1.50	.17	.11	84.69	9.53	4.70
14	I super	14.40	1.41	.37	1.03	.00	.11	85.26	9.63	4.50
	Long pepper:									
15	No. 1, H	12.15	13.48	2.28	5.52	5.68	.53	58.98	8.29	1.71
16	No. 2, T	14.93	11.98	2.37	5.83	3.69	.83	46.16	8.52	1.70
17	Black pepper husks	12.37	11.90	2.12	6.37	3.41	.48	41.71	13.81	4.84
18	Do., with same whole	12.60	9.04	3.00	4.12	1.92	.02	47.36	13.07	4.10
19	Sifting before grinding	7.96	51.39	1.02	6.47	43.90	.80	30.66	7.52	1.15
20	Black pepper, for sale of which fine was inflicted	11.12	14.70	2.02	4.07	8.61	.80	35.85	11.57	2.02
21	Poivrette used to mix with pepper	8.52	3.85	.96	1.05	1.84	0.21	0.00	2.31	.....
22	10 per cent. poivrette, 90 per cent. No. 4	13.34	5.04	2.88	1.78	.38	1.14	49.98	......	.....
23	30 per cent. rice, 70 per cent. No. 4	12.76	3.10	1.68	1.39	.03	.89	88.21		....

Of the above samples Nos. 1 to 5 and 7 to 8 were black pepper corns ground just as imported. The uniformity in the starch in all these lead Heisch to believe that any result under 50 per cent. should be regarded as suspicious, it being very easy to detect the addition of foreign starch, such as rice. Of the white peppers the first three are white pepper corns ground as imported, the next three black corns decorticated in England and then ground.

Of the long pepper Heisch says the starch is double the size of the ordinary and much more angular and like rice. Care must be taken therefore not to confound them. The poivrette is made from olive stones.

The figures are of interest and would seem to confirm the work of Lenz.

Prof. J. Campbell Brown has devoted much time to the subject of pepper and has very recently called attention in England to an adulterant which first made its appearance in Liverpool last summer and since then has been often met with. It is known as pepperette or poivrette, and proves to be the same adulterant so often mentioned in France, olive stones. It resembled walnut shells and almond shells somewhat, but olive stones more so.

Brown gives the following analyses and says:*

	Ash.	Matters soluble by boiling in diluted acid.	Albuminous and other matters soluble in alkili.	Woody fiber, insoluble in acid and alkali.	Starch.
White pepperette	1.33	38.32	14.08	48.48	None.
Black pepperette	2.47	34.55	17.66	47.69	None.
Ground almond shells	2.05	23.53	24.79	51.68	None.
Ground olive stones	1.61	39.08	15.04	45.38	None.

The stones of olives, imported in pickle for table use, gave 3.68 per cent. of ash, but well-washed olive-stones, thoroughly burnt to a white ash, gave under 2 per cent. of ash-like poivrette. "White poivrette" is therefore cleaned very pale, and perhaps partly bleached olive-stones or precisely similar tissue; black poivrette is the same, mixed with a little black husk. It is to be noted that although it contains no starch, yet it yields some sugar to Fehling's solution, after being boiled for some time with dilute hydrochloric acid. The quantity depends on the length of time and strength of acid, but may be stated approximately about 10 per cent. It is important to bear this fact in mind when making a full chemical analysis of pepper containing poivrette. After removing from such a mixture the matters soluble by boiling in dilute caustic alkali, the woody fiber which remains has a yellow color. It consists of the poivrette and some of the cells of pepper husk and one of the subcortical layers of the pepper berry. The pepper cells are made lighter and the poivrette cells darker by the alkali, so that the two are more nearly of a similar yellow color after treatment with alkali. This renders it more difficult to distinguish such of the cells as have somewhat similar markings, but it enables us to distinguish more clearly as poivrette the many torn particles which have no definite form or markings. The final examination of the complete cells is better made with good daylight rather than with artificial light, and in a portion which has been treated with water only.

The pepper cells are mostly different in shape and are colored, and have generally a dark substance in the interior. They are not numerous, but the quantity varies in commercial samples, owing to the modern practice of decorticating the pepper berry to every different extent possible, and mixing the various portions so obtained, including husks, in every variety of proportion with each other or with ordinary pepper. Each individual analyst must make himself familiar with both kinds of cells, as no description can convey an adequate idea of either. In order to form a judgment regarding the proportions of the different chemical constituents of commercial samples, we require to know the chemical composition of the different layers of the pepper-corn, and I hope soon to communicate to the society some figures bearing on this point, as well as to notice some other substances used in the sophistication of pepper.

It is interesting to note that the exemption, mentioned in section 8 of the sale of food and drugs act, in the case of a label being affixed to the article sold intimating that the same is a mixture, does not apply in the case of poivrette, the admixture being made manifestly for the purpose of fraudulently increasing the weight and bulk.

In a subsequent note Brown † warns analysts not to confuse an excessive amount of cortical cells of the pepper husk for poivrette, as they are somewhat similar. This, however, would certainly not occur if authentic samples of pepper and olive-stones were used for comparison.

Brown also contributes a paper to the Analyst § on the use of "long pepper," *Chavica Roxburghii*, as an admixture to the ordinary ground

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* Analyst, **12**, 23-25.

† Analyst, **12**, 47-48.

§ Analyst, **12**, 67-70.

article, showing that it should be discouraged, from the fact that brings with it a large amount of dirt and mineral matter, and has a disagreeable offensive odor developed by warmth. Brown says:

> It is now time that all should take up a decided position in regard to this form of adulteration. Long pepper is the fruit of *Chavica Roxburghii*,* and does not consist merely of the berries analogous to the pepper-corns of the true pepper-plant; it bears much the same relation to them that wild grass-seed would bear to oatmeal. It consists of the small berries with the husks and indurated coverings hardened together and to the central woody stem, much in the same way that in pines the seed and coverings are all hardened into one cone. Long pepper is for the most part derived from wild plants of *Chavica Roxburghii*, which grow by the sides of the water-courses in India. Consequently it always brings with it a mass of dirt, picked up from the soil of the banks whereon it grows, embedded in the crevices and irregularities of the fruit, which dirt the native collector takes care not to lessen, but rather to increase, seeing that he is paid by weight for what he brings down to the merchants.
>
> In commerce we find accordingly that it has always from 3 to 7 per cent. of insoluble sand and clay, in addition to the proper ash of the fruit. And it is difficult, if not impossible, to clean long pepper before grinding in the way that true pepper can easily be cleaned; it can with difficulty be cleaned by hand.
>
> The ash contains a very large proportion of salts insoluble in hydrochloric acid. When ground, the hard husk and woody center, as well as the dirt, are necessarily ground along with the minute berries. The ground long pepper contains not only sand, but more woody fibre than ground genuine pepper of the corresponding shade, although not so much total cellulose as the most husky black pepper. It has the composition shown by Mr. Heisch in his paper. I can confirm his results by the following:

Analyses of long pepper carefully cleaned by hand.

	Total a	Sand and ash insoluble in HCl.	Total matter soluble in 10 per cent. HCl.	Starch and matters convertible into sugar.	Albuminous matter soluble in alkali.	Cellulose.	Extracted by alcohol.	Extracted by ether.	Total nitrogen.
1	8. 91	1. 2	67. 83	44. 04	15. 47	15. 70	7. 7	5. 5	2. 1
2	8. 98	1. 1	68. 31	49. 34	17. 42	10. 50	7. 6	4. 9	2. 0
3	9. 61	1. 5	65. 91	44. 61	15. 51	10. 73	10. 5	8. 6	2. 3

Although the cost of long pepper is at present nearly as high as some very inferior varieties of black pepper, yet the price is generally decidedly lower; even now long pepper is much cheaper than the pepper with which it has been sometimes mixed of late, and its use affords a handsome illegitimate profit, to the detriment both of the grocer and his customer. Long pepper has been, and is, legitimately used for pickles, but it is not known, nor has it been recognized by the trade, as ground long pepper; and all the respectable grocers, and others of whom I have inquired, say decidedly that they would not buy, nor retain, if received, any ground pepper which they knew or suspected to contain an admixture of long pepper. In fact, it is no more right to give pepper containing long pepper in response to a request for simply "pepper" than it would be to give horse-chestnuts instead of Spanish chestnuts in response to a request for simply "chestnuts." It may, of course, be sold as ground long pepper without offense, but no one would buy it. Not only is long pepper a fraudulent admixture in ground pepper, but it is objectionable on the score of quality and flavor. Its disagreeable, offensive odor is developed by warmth. Any candid person can convince himself of the real cause of the objections which housekeepers and grocers

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* Known also as *Piper officinarum* and *Piper Longum.*

alike have to ground long pepper if he will heat up a piece of cold meat between two plates and sprinkle some fresh long pepper on it; the smell and flavor are so offensive that he will feel obliged to reject the meat.

Much of that which one gets whole in shops is very old, and has lost much of its flavor and strength, so small a sale does it command.

The presence of long pepper in ground pepper may be determined by the following characters:

1. *Color.*—If any serious quantity of long pepper is ground in with the ordinary pepper it imparts some of its peculiar slaty color; but this is made much lighter by the now very common practice of sifting out much of the darker or husky portions of the long pepper before mixing it with the genuine pepper. Bleaching is also resorted to, but not hitherto very effectively.

2. The odor of the mixture when warmed is unmistakable by an educated olfactory sense, even if the quantity is comparatively moderate. Attempts are made to disguise the odor by bleaching, but this has not been successful. The ethereal extract also, and even the alcohol extract from which the solvent has been evaporated at a low temperature, yields, when warmed, the characteristic odor very plainly.

3. Long pepper introduces sand into the pepper with which it is mixed, often to a considerable amount. If the pepper is white, this has more importance than has hitherto been accorded to it; for white pepper does not contain, even as imported, 2½ per cent. of sand, and any white pepper containing so much sand must have had the sand improperly introduced, either by direct mixing of Calais sand or in some other way.

Long pepper from which the husk particles have been sifted out when added to white pepper invariably introduces its sand along with it, as well as some spent bleach, if attempts have been made to bleach it.

4. The woody matter in ground long pepper is always considerable, arising both from the smallness of the berries, compared with the hardened setting, and from the central woody tube. This may be detected either by chemical analysis or by the microscope, and some of it by the naked eye or a large hand lens.

If the sample is spread out in a smooth thin layer on strong paper, by means of an ivory paper-knife, pieces of fluffy woody fiber will be detected, especially if the smooth thin layer be tapped lightly from below. Those pieces come from the central part of the indurated catkin which cannot be completely ground fine as genuine pepper stalks are, and are very characteristic if carefully examined. Much of these are of course removed by the grinders' sieves, but enough finds its way through the meshes of the silk to be useful as a corroborative indication.

5. Particles of husks, if present, can be distinguished from genuine pepper husks.

6. A proportion of the starch granules of long pepper are of larger size, above .0002 inch, and of angular shape, very slightly smaller than rice granules, and more loosely aggregated in clusters or isolated.

Brown also calls attention to the statement of authorities that genuine pepper starch is round in form, and shows that this is not always the case. By reference to our illustration, Fig. 65, Plate XXVIII, it will be seen that he is correct. He has lately found* that Dhoura corn, a variety of sorghum, is being largely used in England as a diluent of pepper. The grain is well known in this country as Egyptian corn, and is a common crop in the South and Southwest, but has not been used here as an adulterant. Brown says:

I have met with it only about four times in pepper, but it probably occurs more frequently in other districts. It is known in England as great millet or Turkish

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* Analyst, **12**, 89–90.

millet, and is the grain (with an integument, but without the husk) of one of the cereal grasses, *Sorghum vulgare*.

It is a roundish or oval somewhat flattened grain, size from one-eighth to one-fifth of an inch in diameter, white in color and brittle, with a thin, smooth integument or testa, showing under a high microscopic power, on the inner surface, an aggregation of very small granules, which become blue by iodine. The body of the seed is very white, and consists mainly of roundish or irregular starch granules, varying in size from .0001 up to .0006 of an inch in diameter, and showing under polarized light a nearly right-angled cross; and of larger irregularly rounded granules of starch from .0005 up to .0013 of an inch in diameter, showing no cross, or only a very faint one, under polarized light.

Some of the first-named granules have a hilum and star in the centre, somewhat like bean starch. By boiling with caustic alkali the cellular membrane which binds the starch granules together is disclosed.

The influence of an admixture of sorghum with pepper upon analysis of the latter will be seen from the following analysis of sorghum grains:

Moisture 11 per cent.

Composition of the dried sample.

	I.	II.
Ash	1.31	1.69
Soluble in 10 per cent. hydrochloric acid	90.70	87.80
Starch	75.20	73.00
Albuminous matters soluble in caustic alkali	6.71	7.96
Cellulose	2.56	4.19
Alcoholic extract	10.36	7.96
Ethereal extract	10.10	7.30
Nitrogen	1.82	1.79

There would be no difficulty in detecting it.

Although as yet these substances do not seem to have reached us as far as the samples which we have examined show they must be carefully watched for.

In this country considerable has been published as to the adulterations of pepper, but little in regard to the manner of detecting them. We have already spoken of the large numbers of samples which have been examined in different years by the public analysts of Canada and smaller numbers by those of Massachusetts and New York. Reference to the reports of the department of inland revenue of the Dominion (supplements on adulteration of food) shows that wheat flour, husks, cayenne, coacoanut shells, and pepper dust, milling refuse, pea meal, and sand and clay are in very common use. In the United States the samples examined have apparently proved no better, for while in Canada in 1885 twenty-nine out of sixty samples were adulterated mostly from 10 to 20 per cent., but reaching 75 per cent.; in New York in 1882, out of forty-seven, thirty-three were adulterated, and in Massachusetts in 1884 Wood found one hundred and four in one hundred and ninety-nine impure, and in 1883 69 to 70 per cent. were bad.

We are thus supplied with considerable experience in the examination of peppers, the results of which furnish the basis of a scheme for general use. Thus in examining a sample I should propose to proceed as follows:

METHOD OF EXAMINING PEPPERS MICROSCOPICALLY.

As a preliminary the sieve examination, already mentioned, is of value, the coarser particles left upon a 40 or 60 mesh sieve frequently revealing the nature of the adulterant or the too large proportion of pepper husk. Afterwards it is well, with a good dissecting microscope and a power of 15 to 30, to sort over the ground pepper, and judge of the frequency of the occurrence of the coarse particles, which after a little experience there will be no difficulty in doing. The presence of sand or a notable excess of P. D. can also be detected and estimated in this simple way. Backgrounds of white and black, with reflected light and afterwards transmitted light, may be used in the manner so conveniently afforded by Zeiss's stand, made for this purpose.

A portion of the powdered pepper or the separated coarse particles should also be treated with chloral-hydrate solution for twenty-four hours, to render it more transparent for examination with higher powers, and in the mean time the coarse particles sieved from the powder may be examined under a one and a one-half inch objective, and then crushed and re-examined, using both plain and polarized light. In this way husky matter may be distinguished and foreign starches detected. Polarized light is then the means of bringing out more plainly the starches, the proportion of which iodine will reveal, making due allowance for the small granules of pepper starch and all optically active tissue, such as the bast fibers and sclerenchyma or stone cells, which are found in olive-stones and cocoa-nut shells.

The structure of the pepper itself, as has been explained, is so characteristic as not to be readily confused with foreign matter. In the chloral hydrate preparation, which should now be examined, much of this disappears as the starch is much swollen by this reagent. The husky matter present is rendered thereby so much clearer on the other hand that its identification and differentiation is made much easier, and it is here that the possibility of fixing the source of the adulterant will often lie.

Experience with a half dozen samples from a cheap grocery in comparison with a laboratory sample of pure pepper will soon teach one the best means of making out what has been briefly described.

It has also been found most valuable to digest about a gram of pepper with nitric acid, sp. gr. 1. 1, and chlorate of potash for several hours, or until the color is bleached. It is then possible to distinguish the denser cellular structure more easily than in any other way, particularly the stone cells which make up the larger part of the cocoanut shells and ground olive stones, especially with polarized light, being careful not to confuse the stone cells of the pepper husk with those of olive stones or other adulterants. Charcoal at the same time remains unbleached.

To determine the merits and correctness of the various chemical processes and statements in regard to them previously referred to, a series of pure whole peppers, direct from importers, and of the commercial ground article, have been collected. The results also reveal the extent and nature of the adulteration practiced in this part of the country. The specimens were of the origin described below.

Sources of specimens of pepper.

WHOLE PEPPERS.

No.	Kind.	Remarks.
4514	Whole black	First quality, probably west coast.
4840	do	West coast, direct from importer.
4894	do	Aachen, direct from importer.
4895	do	West coast, direct from importer.
4896	do	Singapore, direct from importer.
4516	Whole white	First quality, source doubtful.
4898	Do	Singapore, direct from importers.

GROUND PEPPERS.

No.	Kind.	Remarks.
4515	Black	First quality, grocers' guaranteed pure.
4523	do	Ground in Washington.
28	do	Ground in Baltimore.
33	do	Do.
37	do	Do.
43	do	Ground in London, guaranteed
52	do	Ground in Washington.
53	do	Do.
4883	do	Ground in Baltimore, low grade "Best."
4884	do	Ground in Baltimore, low grade "Pure."
4524	White	Ground in Washington.
4544	do	Ground in London, "Pure."
4555	do	Ground in Washington.
4882	do	Ground in Baltimore.

MECHANICAL AND MICROSCOPICAL EXAMINATION.

The weight of the whole peppers and the amount of dirt present, as they are imported, have been given already. In the ground condition they of course displayed the normal structure of the berry, as has been already described. No further reference is necessary, therefore, under this head. In the ground specimens the following peculiarities were noted.

4515. Sifting reveals the presence of pepper stems which should not be present, showing that the pepper was ground without cleaning and an undue proportion of husky matter, unbleached by nitric acid and chlorate.

4523. This specimen is very coarsely ground, a large proportion of husk remaining on the 40-mesh sieve, among which evidence of the presence of maize could be distinguished, and that P. D. in some form must be present.

4528. Sifting separated light chaffy and fibrous matter. The microscope detected yellow corn and its hulls.

4533. This proved of very bad quality, the siftings consisting of bran, roasted shells or charcoal, and corn. The microscope was confirmatory, and the presence of the roasted shells prevented bleaching with Schulze's reagent. This is evidently a P. D. pepper of the worst sort.

4537. Sifting and examination showed the presence of P. D. in some form and corn. It contains no roasted matter or charcoal.

4543. Proved to be quite pure and well ground, all the material passing a 40-mesh and nearly all a 60-mesh sieve. This is the only *pure ground* sample met with.

4552. Sifting shows the presence of a complicated collection of adulterants, husks of various origin, &c. Microscopic examination detected mustard hulls, corn, roasted shells, or charcoal not bleachable, and other foreign material not identified.

4553. Contains mustard hulls and branny matter, but no charcoal. *Bad.*

4883 and 4884. From the same mill in Baltimore were the worst specimens met with. They contained but little pepper and were made up of P. D., yellow corn, cracker dust, cayenne, charcoal, and other foreign matter.

4882. A white pepper from the same source was of the same origin, leaving out the black elements.

4524 and 4544. These white peppers were found to be pure, but the former not carefully decorticated.

4555. Contained foreign starchy matter and probably cayenne. Sifting revealed nothing abnormal.

These examples serve to show the variations which are met with and what the analyist may expect. It is always well, also, to be on one's guard for something new.

As a confirmation of the physical examination and a means of determining the amount of adulteration in the several cases determinations were made of the proximate composition:

Analysis of peppers.

Serial number.	Source.	Per cent. of dust.	Weight of 100 grains.	Quality.	Water.	Ash.	Volatile oil.	Piperine and resin.	Alcohol extract.	Starch.	Undetermined.	Crude fiber.	Albuminoids.	Total.	Total N. ×6.25.	Total N.	Reducing sugar equivalent, on dry ash free pepper.
	[Whole black.]																
4514	West Coast	Clean	5.900	Cleaned	8.91	4.04	.70	7.29	......	36.52	24.62	10.23	7.69	100	9.81	1.57	47.16
4840	......do	Clean	6.460	Retail	8.15	2.91	1.48	7.20	5.08	33.02	21.02	8.74	11.50	100	13.65	2.18	42.38
4894	Acheeu	2.5	4.525	As imported	8.29	4.70	1.69	7.72	6.06	37.50	13.64	10.02	10.38	100	12.60	2.02	47.87
4895	West Coast	4.3	5.085	......do	9.36	4.52	1.63	7.90	5.71	36.18	13.59	10.30	10.81	100	13.13	2.10	46.68
4896	Singapore	8.3	4.870	......do	9.83	3.70	1.60	7.15	5.74	37.30	17.66	10.02	10.00	100	12.08	1.93	44.13
	[Whole white.]																
4516	West Coast	Clean	5.130	Retail	9.85	1.41	.57	7.24	......	40.61	23.28	7.73	9.31	100	11.48	1.83	50.86
4898	Singapore	1.4	4.960	Imported	10.60	1.34	1.26	7.76	2.57	43.10	19.55	4.20	9.62	100	11.90	1.90	54.38
	[Commercial ground black.]																
4515	Washington grocers			Adulterated	10.00	4.52	.69	6.50	......	31.86	21.53	15.90	9.00	100	10.88	1.74	39.80
4523	......do			......do	11.83	4.38	1.04	6.59	......	21.56	23.49	10.55	20.56	100	21.88	3.50	28.52
4528	......do			......do	12.30	6.54	.84	5.94	......	30.00	18.94	15.93	9.51	100	11.20	1.79	40.90
4533	......do			......do	8.92	4.06	.92	3.99	......	30.83	21.52	20.48	9.28	100	10.33	1.65	39.36
4537	......do			......do	11.15	7.85	.82	6.59	......	27.85	17.30	19.03	9.31	100	11.20	1.79	38.19
4543	......do			Good	10.70	5.85	.42	7.19	......	30.47	20.81	14.90	9.66	100	11.73	1.88	40.57
4552	......do			Adulterated	7.60	7.50	2.94	5.02	......	27.04	25.55	11.20	12.25	100	13.95	2.23	35.40
4553	......do			......do	8.90	9.85	1.14	6.38	......	21.27	27.13	13.53	14.80	100	16.63	2.66	29.08
4883	Baltimore Mills			......do	9.48	6.15	2.09	2.66	6.54	28.19	14.83	18.13	11.93	100	12.60	2.02	37.13
4884	......do			......do	8.45	5.29	1.66	2.80	5.61	31.34	16.36	16.73	11.73	100	12.43	1.99	40.37
	[Commercial ground white.]																
4524	Baltimore Mills			Pure	11.56	1.16	.48	7.11	......	41.00	25.30	6.13	8.20	100	10.33	1.65	52.28
4544	......do			......do	11.35	1.86	.55	7.45	......	48.29	14.35	4.63	12.52	100	13.80	2.20	59.81
4555	......do			......do	8.95	1.35	1.25	7.22	......	50.30	16.55	5.98	8.40	100	10.50	1.68	65.73
4882	......do			Adulterated	11.12	2.13	.08	2.81	4.10	15.58	4.24	12.13	10.79	100	11.55	1.85	66.09

The analyses of the pure specimens of pepper show that in all the amount of water was between 8 and 10 per cent. This is of course variable with surrounding conditions. The ash in black peppers did not exceed 4.7 per cent., and in white 1.4. It is fair to believe that anything above 5 per cent. for black and 2 per cent. for white is suspicious, otherwise adulteration or dirt are certainly present.

The volatile oil varies in black pepper from 1.69 to .70, and in white 1.26 and .57 were found. This determination is not of great value as a means of detecting adulteration. Piperine and resin, however, furnish a most valuable check on the purity of the samples. In the whole berries ground by us both black and white contained from 7.90 to 7.24 of these substances, showing great constancy in amount, and on the addition of adulterants this is plainly affected. It serves, in my opinion, as well or better than determinations of pure piperine, the latter being difficult and involving loss, as has been shown by careful experiments made by Mr. Knorr under my direction. It has also proved impossible to make determinations of piperine by the combustion or Kjeldahl methods by application of Stützer's copper hydrate process, the per cent. of nitrogen being so small, 4.912 in piperine, as to make the error very large when converting the former to the latter, the necessary factor being 20.36. Rottger's objection to the value of the determination of the ether extract is not sustained in our experience, and it seems that he must have employed inferior ether, which is often the cause of serious error. Only the best Squibb's ether or its equivalent should be used for extractions.

The alcohol extract appears to be of no value.

The determination of starch or its equivalent in reducing sugars has been looked into with care and the conclusions arrived at are that the results are of value when carried out in the manner which has been described under the general head of methods of analysis. A preliminary extraction with alcohol and water is necessary to obtain results which are fairly constant. Determinations made in this way show that black peppers contain from 34 to 38 per cent. of starch, or 42 to 47 per cent. of substances of reducing sugar equivalent calculated on dry ash free substance. White peppers contain in the same way from 40 to 43 per cent. of starch, and give from 50 to 55 per cent. of reducing sugar equivalent on dry ash free substance. These figures are not as high as those given by Lenz, but are the result of careful work on pure samples, extending over a long time and involving much experience. It is believed that they must be nearly correct. In our opinion there is no advantage in calculating the results to a sugar equivalent unless excessive moisture or ash is found to be present.

The crude fiber in the black peppers, as determined by our methods, does not vary far from 10 per cent. One sample contained but 8.74, and of course in the white peppers the amount is much reduced, depending to a certain extent on the perfection of the decortication; 4 to

8 per cent. are probably fair limits. The determination is of comparative value, revealing the presence of foreign woody or fibrous matter.

The albuminoids do not vary widely, 10 per cent. being the average, with extremes in our experience of 7.69 and 11.50. The addition of nitrogenous seeds of course increases the amount, and of fibrous or woody matter diminishes it. As will be seen, therefore, the determination is a useful one. The variations, then, may be summarized as follows:

	Black.	White.
Water	8.0 to 11.0	8.0 to 11.0
Ash	2.75 to 5.0	1.0 to 2.0
Volatile oil	.50 to 1.75	.50 to 1.75
Piperine and resin	7.0 to 8.0	7.0 to 8.0
Starch	32.0 to 38.0	40.0 to 44.0
Crude fiber	8.0 to 11.0	4.11 to 8.0
Albuminoids	7.0 to 12.0	8.0 to 10.0

With these pure peppers the ground samples must be compared. In the table the determinations which are suspicious are printed in full-faced type. All but one of the black peppers are convicted of adulteration on the chemical evidence.

The first, 4515, has too little piperine and resin, too little starch, and too much fiber. It is apparent that some fibrous or woody diluent has been added, as appears from the microscopic examination to be true.

Without discussing each analysis in detail it may be pointed out that only three samples contained an excess of ash, as a rule being free from mineral adulterants, and the three cases are probably only dirty, so that the addition of minerals is not common in this country.

The piperine and resin were deficient in all the specimens except the one pure one, revealing at once sophistication.

Starch was deficient in all the specimens, in the pure specimen falling below the usual limits, which at the same time was more than usually husky, thus accounting for the difference, and in fact in all but two cases there was present more fiber in the peppers than we have considered normal, another indication of the presence of adulterants or dirt.

The albuminoids in two cases were present in excessive amount, revealing the presence of some foreign seed rich in nitrogen. In the other specimens the adulterants did not throw this determination far away from the normal.

In the white peppers the presence of an excessive amount of starch in 4555 makes it suspicious, and the great deficit in piperine and excess of starch and fiber show that 4882 is adulterated.

The value of the chemical determinations is thus plainly illustrated. Perhaps with no one of the substances which are considered in this report are the indications more certain. It would be possible, too, in an indirect way, by means of proportions such as have been already mentioned, to calculate with an approximation to accuracy the extent of the adulteration. It is fortunate that in a material which is probably more frequently adulterated than any other the presence of foreign matter is so easily detected.

CAYENNE.

Cayenne or red pepper is the powdered pod of several species of *Capsicum*, a genus of the family *Solanaceæ*, to which the potato and tomato belong, the commonest species being *C. annuum* and *C. fastigiatum*, known also under many synonyms.

The first, Flückiger and Hanbury state, furnishes the larger kinds of pod pepper, and, as they believe, much of the cayenne pepper which is imported into England in a state of powder.

C. fastigiatum is the species which is officinal in both the British and United States pharmacopœias. It is grown in tropical Africa and America and appears in our market as Zanzibar pepper. The color of its powder is lighter yellow than that of the preceding. The two species furnish the market with its cayenne pepper, although a few other species are sought in small amount for their peculiar flavor.

Of the microscopic structure Flückiger and Hanbury say:

> The pericarp consists of two layers, the outer being composed of yellow thick-walled cells. The inner layer is twice as broad and exhibits a soft shrunken parenchyme, traversed by thin fibro-vascular bundles. The cells of the outer layer especially are the seat of the fine granular coloring matter. If it is removed by an alcoholic solution of potash, a cell nucleus and drops of fat or oil make their appearance. The structural details of this fruit afford interesting subjects for microscopical investigation.

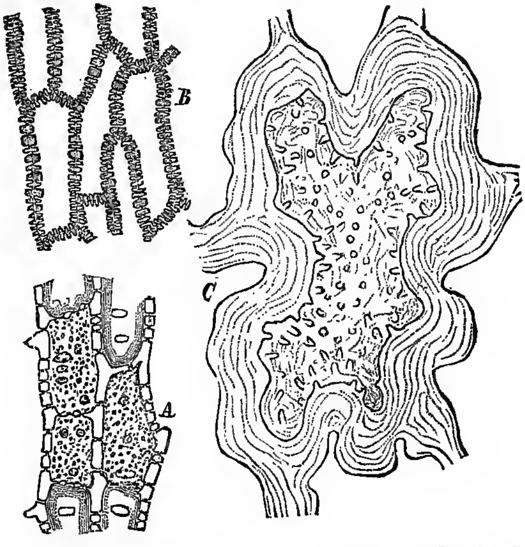

FIG. 12. Cayenne; *A*, outer epidermis. *B*, stone cells of the inner epidermis; *C*, epidermis cells of the edge of seed. (After Schimper.)

The peculiarities described above are so distinctive that the presence of foreign matter is easily detected. The cells of the pericarp or epidermis are of a peculiar flattened and chain-like angular form which are characteristic of cayenne. The other structures are not as prominent, but are not liable to be confounded with those of any adulterants. Diagrammatic representatives of this structure are given in Fig. 12, and the appearance of the pure ground cayenne under polarized light in Fig. 48, Plate XXIV. The portions of the seed in the powder are not readily distinguished without careful examination. They are, however, characteristic and contain starch, the form of which is shown in Fig. 67, Plate XXVII. The adulterants, which are said by Hassall to be more numerous and frequent than with ordinary pepper, are given by authorities as mineral coloring matter added to hide the loss of color which takes place on exposure of cayenne to light, to add to the weight, or to cover the addition of colorless diluents, ground rice, turmeric, husks of mustard, etc.

Mineral adulterants were met with in but one sample of a low grade, obtained in the Baltimore market. The organic ones, on the contrary, were almost always present, among them yellow corn meal being found, but rice or corn flour oftener. The latter was readily detected with the polariscope, as illustrated in Fig. 49, Plate XXIV. The few starch grains in the lower layers of the pericarp and in the seed are very small and cannot be confused with the added rice or corn. Turmeric and mustard are recognized by their peculiar structure.

Of the chemistry of *Capsicum* but little had been written until 1884, when Strohmer undertook an investigation of the proximate composition of the pod. Bracconot had announced the discovery of the active principle, and named it capsicine. Wilting had asserted that it was a crystalline alkaloid. Thresh had discovered a crystalline body capsicine, which he regarded as the substance to which the pungency was due, but Strohmer showed that there was present in the cayenne—

(1) A fixed oil, without sharp smell or taste, that required 201.9 of KOH for saponification and occurred almost entirely in the seed.

(2) A camphor-like substance which tastes and smells sharp, and which constitutes the peculiar principle of the cayenne (capsicine). It is contained in the pods and seeds, although in greater amount in the former than in the latter, where it is dissolved in a fixed oil.

(3) A resinous body, the red coloring matter, (capsicum red) which is only contained in the pod.

Quantitative determinations were made of the fruit of *Capsicum annuum*, grown in Hungary.

	Seed.	Pod.	Whole fruit.
Water at 100°	8.12	14.75	11.94
Albumineids	18.31	10.69	13.88
Fat. (ether extract)	28.54	5.48	15.26
Nitrogen free extract by difference	24.33	38.73	32.63
Crude fiber	17.50	23.73	21.09
Ash	3.20	6.62	5.20
Total	100.00	100.00	100.00
Nitrogen	2.93	1.71	2.22

The percentage of water given is probably too high, owing to the volatilization of the camphor-like substance.

The ether extract of the seed was nearly pure fat, that of the pod capsicum red.

As to whether a pepper is adulterated or not, Strohmer considers that these determinations will furnish the evidence. He found in commercial Cayennes:

	(I) Rosenpaprika, prima.	(II) Rosenpaprika, sekunda.	(III) König's, paprika.
Water	17.35	14.39	12.69
Albuminoids	14.56	14.31	13.19
Ether extract	14.43	15.06	13.35
Ash	5.10	5.66	7.14

The latter specimen was adulterated with stems, &c., and the results show the agreement between the degeneration in quality and the analytical determinations.

In our investigations several specimens have been examined with the following results:

4517. English brand.
4545. New York brand.
4554. Bulk, corner grocery.
4554.^{1} Public lunch-room.
4880. Baltimore spice mills.
4881. Baltimore spice mills.
4897. Zanzibar; pure, whole, from importers.

Microscopic examination.

4517. Pure, Crosse and Blackwells. Bottled.
4545. Adulterated rice flour.
4554. Adulterated rice flour.
4554.^{1} Adulterated rice flour.
4880. Adulterated } yellow corn, turmeric, and red ocher.
4881. Adulterated }

The analytical results were:

Serial No.	Water.	Ash.	Fixed oil.	Volatile camphor, &c.	Fiber.	Albuminoids.	Undetermined.	Total.	Nitrogen.	Quality.
4897	2.35	9.06	0.12	26.99	16.88	13.13	41.47	100.00	2.10	Pure.
4517	5.74	5.24	1.58	17.90	18.10	11.20	40.24	100.00	1.79	Do.
4545	3.70	6.10	3.40	14.50	15.40	10.15	46.75	100.00	1.62	Adulterated.
4554	5.20	6.65	1.65	14.11	10.53	9.98	51.88	100.00	1.60	Do.
4880	1.41	4.68	4.00	9.41	14.70	7.70	58.10	100.00	1.23	Do.
4881	1.93	8.69	3.48	6.66	13.33	7.00	58.91	100.00	1.12	Do.

From the data it is not difficult to detect the presence of adulterants. The whole Zanzibar pepper ground in the laboratory has 26.99 per cent. of non-volatile ether extract, much more than was found by Strohmer, and the other pure specimen 17.90 per cent. None of the others reach these figures. In albuminoids the addition of starchy and mineral diluents also makes a marked difference, their presence being revealed in a striking way.

The unexplained presence of so much volaltile matter on the two worst specimens and of so much ash in the Zanzibar of good grade as compared with these same specimens containing mineral adulterants, is remarkable and shows that too hasty conclusions must not be drawn from the chemical data. It should be noticed also that the water in our specimens falls far below that given by Strohmer, a peculiarity which has been noted in other cases. In the detection of adulteration of Cayenne by chemical methods, determinations of water and ash, ether extract and albuminoids are, it seems, of value and as a rule will serve to reveal the means of adulteration. When combined with a microscopic examination the estimation of the amount of the diluents present would not be difficult.

GINGER.

The rhizome, or commonly root, of *Zingiber officinale* is known as ginger. It is a "reed-like plant, with annual leafy stems 3 to 4 feet high, and flowers in cone-shaped spikes borne on other stems thrown up from the rhizome. It is a native of Asia, in the warmer countries of which it is universally cultivated, but is not known in the wild state. It has been introduced into most tropical countries, including the West Indies."

Ginger occurs in two forms—dried with the epidermis as coated ginger and as scraped ginger when the epidermis is removed. The uncoated ginger is prepared by scraping and washing the rhizome and then drying it in the sun. Thus prepared, Flückiger and Hanbury say:

> It has a pale buff hue and breaks easily, exhibiting a short and farinaceous fracture with numerous bristle-like fibers. When cut with a knife the younger and terminal portion of the rhizome appears pale yellow, soft, and amylaceous, while the older part is flinty, hard, and resinous.
>
> Coated ginger or that which has been dried without the removal of the epidermis is covered with a wrinkled, striated brown integument, which imparts to it a somewhat coarse and crude appearance, which is usually remarkably less developed on the flat parts of the rhizome. Internally it is usually of a less bright and delicate hue than ginger from which the cortical part has been removed. Much of it, indeed, is dark, horny, and resinous.

In our markets we find Jamaica ginger, which is the finest variety, being very carefully prepared and scraped, and several qualities of brown ginger which is unscraped and not carefully prepared, and is imported from India and the East. The latter is sometimes bleached and coated with gypsum or carbonate of lime to improve its appearance. This method of treatment appears to be very common.

Of the microscopic appearance of the rhizome Flückiger and Hanbury write as follows:

> A transverse section of coated ginger exhibits a brown, horny external layer, about one millimetre broad, separated by a fine line from the whitish mealy interior portion, through the tissue of which numerous vascular bundles and resin cells are irregularly scattered. The external tissue consists of a loose outer layer, and an inner composed of tabular cells; these are followed by peculiar short prosenchymatous cells the walls of which are sinuous on transverse section and partially thickened, imparting a horny appearance. This delicate felted tissue forms the striated surface of scraped ginger and is the principal seat of the resin and volatile oil, which here fill large spaces. The large-celled parenchyme which succeeds is loaded with starch and likewise contains numerous masses of resin and drops of oil. The starch granules are irregularly spherical, attaining at the utmost 40mm. Certain varieties of ginger, owing to the starch having been rendered gelatinous by scalding, are throughout horny and translucent. The circle of vascular bundles which separates the outer layers and the central portion is narrow and has the structure of the corresponding circle or nucleus sheath on turmeric.

In the best ground ginger of the trade little is seen of this structure. The appearance is one of predominating starch grains which are so abundant in the parenchyma, and which are figured in Plate XXI, Fig. 41, and Plate XXVII, Fig. 58. They are so characteristic in shape and in their appearance with polarized light, that they are readily distinguished from adulterations.

Among them are seen scattered the fibro-vascular bundles, the turmeric-like resin bodies, and less prominently the oil globules, which are few in number.

When the source of the powder is the ordinary unscraped ginger, the outer horny layer is prominent, but not distinct in its character, and at times when the rhizome has been scalded the starch grains are swollen and the whole structure is more difficult to make out. It is not, however, as a rule, difficult to detect foreign matter in the powder of this spice.

ADULTERANTS.

The principal adulterants are said to be mineral matter, sago, tapioca, flour of rice, wheat, and potato, cayenne, mustard hulls, turmeric, and exhausted ginger. The foreign starches, cayenne, and mustard hulls, are easily detected, but the turmeric cells, from their resemblance to the resin globules of the ginger, are more confusing. The too frequent occurrence of such cells is suspicious. For the detection of exhausted ginger recourse must be had to proximate analysis. The recommendation also of Hassall, to wash away some of the starch, or the use of a sieve, will be found of advantage, the remaining coarse particles being then seen in greater numbers and with more ease. We have found in the gingers of the trade rice flour, turmeric, and hulls of foreign seeds. But two of the specimens were derived from coated root.

CHEMICAL COMPOSITION.

Owing to the practice of diluting the ground spice with the residues from extraction for turmeric, &c., the normal proximate composition is of importance.

W. C. Young* has examined seven well-authenticated specimens with results which have been reprinted here.

Sources.	Moisture at 100° C.	Ash.	Ash, insoluble in H_2O.	Ash, soluble in H_2O.	Aqueous extract, 2 per cent. decoction.	Mucilage.	Alcohol extract, 5 per cent. decoction.	Resin.	Cellulose
African 1	15.8	3.4	1.34	2.06	24.8	18.0	8.5	2.2	5.1
African 2	14.5	4.3	1.58	2.72	52.2	......	15.7	..	......
Jamaica	15.0	5.4	1.22	4.18	55.7	32.3	6.5	.25	3.1
Cochin	15.2	5.8	3.28	2.52	35.1	21.8	12.5	4.5	9.0
Japan	15.2	8.0	5.82	2.18	34.3	19.4	8.3	2.8	4.6
Malabar	10.2	3.4	1.6	1.8	30.1	22.4	4.1	1.7	1.7
Bengal	20.50	4.75	2.36	2.39	51.4	41.1	4.3	.84	4.9

These specimens were all scraped with the exception of the last two. The analyses do not seem to have been carried out on accepted principles, and are not of great value except for ash and perhaps fiber, but in complete want of any data they have been copied. The fact that the decorticated specimens contain no more fiber than the others is peculiar. The methods of determining "resin" and "mucilage" and the meaning of the terms also are not given. Little is therefore learned from them.

Tresh† has investigated the ether extract of the rhizome, but his results furnish us with no data of analytical use. Lately E. W. T. Jones‡ has, under the title "The Amount of Starch in Ground Ginger," obtained some data which are of value, but unfortunately are confined to only one specimen of ginger. He determined water and ash as usual, extracted with ether, after moistening with alcohol (Sp. gr 0.90), at 35°–38°, and with water at the same temperature. The residue, after gelatinizing the starch, was submitted to diastatic action. In the filtrate from the unacted-upon substance, which was weighed as fiber, the dextrine and maltose were determined by the polariscope. Fiber was also determined by the usual method of treatment with acid and alkali. The results were:

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*Analyst, **9,** 214–215, 1884.

† Pharm. J., Trans., **37,** 610, 721, 1882.

‡Analyst, **11,** 75–77, 1886.

	Per cent.
Moisture, loss at 100° C	10.10
Ether extract	3.58
Alcohol extract	3.38
Water extract	3.66
Starch, calc., from dextrine and maltose	52.92
Fiber, residue from diastatic action	19.12
Crude fiber by acid and alkali	2.66
Ash	4.80
Matter not accounted for in the starch products	1.50
	99.06

Practically we have learned from all that has been hitherto done in the proximate examination of ginger that the ash may vary from 3.4 to 8.0 per cent. in genuine samples, and fiber from 1.7 to 9.0 per cent. Young and Jones differ altogether too much on other points to be of any value. We are therefore in need of further information to enable us to detect the addition of spent ginger as an adulterant, the differences in determinations of fiber being so wide as to render this determination of no value, since it is possible to even reduce the amount of fiber by such an addition and judicious selection.

With a view to extending our knowledge of the subject we have examined a number of pure gingers, obtained from the importers and several samples of ginger found in the local markets, some of which, as was learned from the microscopic examination, were adulterated with farinaceous matter and foreign hulls. The results were:

Analyses of gingers.

Sources of samples:

4506. Scraped Jamaica, limed, whole.
4889. Whole Calcutta ginger root, unscraped and unbleached.
4890. Whole Cochin ginger root, unscraped and unbleached.
4891. Whole Jamaica ginger root, unscraped and unbleached.
4892. Whole Jamaica ginger root, London market, bleached.
4893. Whole Jamaica ginger root, American market, bleached.
4507. White ground Jamaica, "pure."
4521. Brown, ground in Washington.
4526. Brown, ground in Baltimore.
4541. Genuine Borneo, English brand.
4549. Brown.
4242. Mohawk, N. Y.
4875. Ground ginger, Baltimore Spice Mills, "pure."
4876. Ground ginger, Baltimore Spice Mills, "best."

Microscopical examination:

4506 and 4889–4893. Pure standards.
4507. Not as much fiber matter as in the preceding, but no adulterants detected.
4521. Good brown ginger.
4526. Starch, turmeric, and foreign husks.
4541. Good and white.
4549. Starch, and perhaps turmeric; brown.
4242. Starch and some questionable substance.
4875 and 4876. Cereals and cayenne; perhaps exhausted ginger.

Analyses of gingers.

[Whole ginger root

Serial No.	Source.	Water.	Ash.	Volatile oil.	Fixed oil and resin.	Starch.	Crude fiber.	Albuminoids.	Undetermined.	Total.	Nitrogen.
4889	Calcutta	9.60	7.02	2.27	4.58	49.34	7.45	6.30	13.44	100	1.01
4890	Cochin	9.41	3.39	1.84	4.07	53.33	2.05	7.00	18.91	100	1.12
4891	Unbleached Jamaica	10.49	3.44	2.03	2.29	50.58	4.74	10.85	15.58	100	1.74
4892	Bleached Jamaica, London	11.00	4.54	1.89	3.04	49.34	1.70	9.28	19.21	100	1.48
4893	Bleached Jamaica, American	10.11	5.58	2.54	2.69	50.67	7.65	9.10	11.66	100	1.46
4506	Bleached Jamaica	9.10	4.36	.96	3.09	46.16	3.15	5.25	27.93	100	.84

[Commercial ground.]

Serial No.	Source.	Quality.	Water.	Ash.	Volatile oil.	Fixed oil and resin.	Starch.	Crude fiber.	Albuminoids.	Undetermined.	Total.	Nitrogen.
4507	Bleached	Pure	8.06	3.47	1.78	3.11	48.92	2.65	6.13	25.88	100	.98
4521	Unbleached		11.20	6.02	1.61	4.12	50.00	4.08	6.28	16.69	100	1.04
4526	...do	Adulterated.	10.35	5.83	1.52	4.66	46.49	3.48	8.75	18.92	100	1.40
4541	...do		8.90	3.45	.95	3.65	49.12	3.45	6.30	24.18	100	1.01
4549	...do	Adulterated	9.45	4.75	1.45	4.30	50.58	4.20	8.93	16.34	100	1.43
4875	Brown	...do	11.82	7.34	2.61	3.21	46.49	4.98	7.35	18.20	100	1.18
4876	...do	...do	11.33	7.94	2.11	3.51	49.12	3.20	7.35	15.44	100	1.18

From these results it appears that the ash is quite variable, a pure sample having as much as 7.02 per cent. and as little as 3.44 per cent., the whiter varieties having the least and the brown the most. From the ether extract little can be learned. There is no distinction even between the pure and adulterated, the adulterations having a very similar amount of extract. Of the pure samples the white has the least, as well as the least ash. Of fiber the least, as would naturally be expected, is found in the whitest and best scraped and prepared varieties. Of albuminoids it may be said that there is no greater variation in the pure samples than in those which proved to be adulterated. When foreign hulls are present the amount may be much larger. The determinations of starch do not show that the samples have been diluted with cereals or any farinaceous matter, neither could any be detected under the microscope.

The chemical examination proves consequently most unsatisfactory, and it is only possible to give the following hints for pure ginger:

	Per cent.
Water	11.00 to 9.10
Ash	7.02 to 3.39
Volatile oil	2.54 to .96
Fixed oil	4.68 to 2.29
Starch	53.33 to 46.16
Crude fiber	7.05 to 1.70
Albuminoids	10.85 to 5.25

In our markets ginger is drier than abroad, as is usually the fact with all such substances in our climate. The percentage of ash among Young's samples reached 8.0, but did not go below our lowest determination, and our variations in fiber include his figures and those of Tresh.

In other respects our determinations agree with those of the two investigators mentioned, and from their increased number serve as a fair basis for comparison.

A careful qualitative examination of the character of the extracts at times may reveal the presence of an adulterant, but the chief dependence must, it seems, in the examination of this spice, be placed upon the microscope. This, however, will not reveal the presence of exhausted ginger, and a careful study of the effect of exhaustion on the proximate composition of the ground root is therefore desirable. It would naturally increase the relative of percentages of fiber and albuminoids and starch and diminish that of extractive matter.

CINNAMON AND CASSIA

Cinnamon and cassia are the barks of several species of the genus *Cinnamomum*, the true cinnamon (*C. zeylanicum*) being a native of Ceylon, where it is also largely cultivated, while the cassias are derived from several species growing in Bengal and in the countries east of India, especially China and in the Indian Archipelago. In our markets the Chinese and Malabar cassias occupy the most prominent place, Batavia and various other localities supplying a poorer quality. Ceylon cinnamon is only found in small amount and usually is in use as a drug. In our experience it is not to be procured in a ground condition. In their original form there is no difficulty in identifying the various barks by their peculiar characteristics. Ceylon cinnamon, during its preparation, is deprived of its outer corky coat and of the inner cortical layers. It is therefore thin, not more than one-eighth to one-sixteenth inch thick, and is distinguished by having the quills, which curl inward on each side, arranged one within the other, forming a flattened cylinder, originally a yard long. Its color is a dull brown, and the removal of the outer coats is discovered by the appearance on the exterior of the peculiar wavy structure of the inner layers of the bark.

Cassia, on the other hand, commonly shows the outer corky coat of the bark, and is, in consequence, much thicker, rougher, and of not so red color. In some cases, however, the bark is scraped, but the character of the surfaces are still distinctly marked, and it is thicker and deeper in color than cinnamon.

Microscopically, true cinnamon consists of thin layers, which in the ground drug are represented most prominently by the long cells of woody fiber or liber fibers which are scattered through the bark, and which are distinctly seen under polarized light, the stellate cells of the outer layers and the thinner cells of the interior carrying the few starch grains. In cassia, which of course bears a close general resemblance to cinnamon, the woody fiber is comparatively small in amount, as is the number of the stellate cells. The presence of starch is, on the other hand, more marked. A view, therefore, of a true ground cinnamon and cassia with polarized light reveals at once differences which are characteristic enough to distinguish the specimens, and which are illustrated in figures 46 and 47, plate XXIV. In the ground specimens which we have had in hand, however, no specimens of true cinnamon have been found. Reference was made, therefore, to ground samples prepared from authentic specimens of Ceylon and Chinese barks, and before any microscopic work is attempted it is necessary to make a comparative examination of this description to acquire a knowledge of the appearances to be expected and which cannot be described in detail. Reference may also be usefully made to Flückiger and Hanbury, Pharmacographia, for many details in regard to the growth, cultivation, preparation, &c.

Of the addition of cedar sawdust, roasted hulls, oil meals, and mineral and coloring matter we have had but little opportunity to judge. Adulteration in these markets is mainly confined to replacing cinnamon by cassia, and the adulterants mentioned are found only in the lowest grades, while their entire difference in structure make their detection microscopically a matter of no difficulty.

Schimper in his *Anleitung zur mikroskopischen Untersuchung der Nahrungs- und Genussmittel*, describes in detail the appearance of these foreign substances which in Germany seem to be often used and which Wood in Massachusetts has detected in the forms of ground crackers and nut shells, and in Canada are represented by ground peas and wheat, flour, and, as we have found in Baltimore, by minerals and coloring matter. As will be shown, the determination of fiber and ash are valuable means of discovering the presence of such additions.

CHEMICAL COMPOSITION AND REACTION.

Of the proximate composition of any of the barks but little is known. Numerous determinations and analyses of the ash have been made with a view to detecting peculiarities or the addition of mineral matter.

Hilger* has recently found in five samples of Ceylon cinnamon the following amounts of ash:

	Soluble—per ct.
(1)—4.5	53.0
(2)—4.8	72.3
(3)—3.9	88.1
(4)—4.3	61.7
(5)—3.4	....

Hehner† in 1879 made an extended investigation into the substitution of cassia for cinnamon, and examined the iodine test, finding it useless, as has been the case when employed in our laboratory. He then investigated the mineral constituents, thinking that the more woody cassia

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*Arch. d. Pharm., **223**, 826.

†Analyst, **4**, 223–228.

bark would contain more lime and magnesia than the more delicate cinnamon. Some of his figures are as follows:

Kind.	Price per pound, retail.	Moisture.	Ash in bark.	Lime in ash.	$Mn^3 O^4$.	Soluble ash.	Insoluble ash.
	s. d.						
Whole cinnamon	1 10	12.67	4.78	40.09	.86	25.04	74.96
	3 0	12.05	4.59	36.98	.97	28.98	71.02
	3 6	11.38	4.66	40.39	.13	25.22	74.78
	3 6	11.64	3.44	34.32	.62	26.36	73.64
	8 6	12.94	4.28	36.99	.59	27.67	72.33
Cinnamon chips	*9	11.25	4.44	42.11	.34	18.34	81.66
Cassia lignea:							
Whole		14.22	1.84	25.29	5.11	40.58	59.42
		11.88	2.54	34.49	4.94	26.78	73.22
Ground		11.05	2.55	28.63	3.55	30.91	69.09
Cassia vera		10.37	4.08	52.72	1.13	8.36	91.64
		11.36	4.85	43.40	1.53	15.89	84.11

*Containing wood.

From these analyses Hehner concludes that the ash in cinnamon varies between comparatively narrow limits. *Cassia vera*, or Malabar cassia, contains as much as cinnamon, *Cassia lignea* less; that one quarter of the ash is soluble in water in cinnamon, less in *C. vera*, and still less in *C. lignea;* that cinnamon ash contains less than 1 per cent. of oxide of manganese, *C. vera* more than 1 per cent., and *C. lignea* far more, even up to 5 per cent. This is the most noteworthy feature and is shown in the color of the ash, cinnamon ash being white, while those of cassias are gray or brown. *Cassia vera*, the Malabar cassia, is not so readily distinguished from cinnamon as *C. lignea*, but its occurrence is less frequent. The percentages of lime and magnesia were not distinctive, as had been expected. For cinnamon, 40.09, 36.98, and 40.39 were the amounts of lime in three specimens. In one of *Cassia lignea* 25.29 per cent. was found. In one of *C. vera*, 52.72. The less fibrous *C. lignea* contained the least lime. Of magnesia there was 2.65, 3.30, and 3.86 in the cinnamon and 5.48 and 1.10 in the *Cassia lignea* and *vera* respectively. Little seems to have been learned, therefore, which would form a sound basis for distinguishing these barks. The presence of manganese is more or less accidental and cannot be considered as an essential element of the ash or one from which such definite conclusions could be drawn as to serve as the basis of legal testimony. Our results, in fact, failed to show any large amount in any of the specimens.

Of the proximate organic constituents, the presence of volatile oil, tannin, mucilage, coloring matter, resin, an acid, starch, and lignin is noted by Hassall, without any details in regard to their proportions. The essential oil, according to Pharmacographia, however, amounts to only one-half to 1 per cent. of the bark of cinnamon and must be less in the inferior cassias. Beyond this no data are found. We have examined a number of specimens of the ground bark bearing different designations and three or four samples of bark obtained unground and several authenticated specimens direct from the importers. The sources and descriptions of the material examined were:

DENOMINATED CINNAMON.

4502. Bark from grocers, D. C. Poor quality.
4503. Ground, from same grocers. Both guaranteed, pure.
4519. Ground in Washington, 30 cents per ½ pound.
4529. Ground in Baltimore, 20 cents per ½ pound.
4531. Ground in Baltimore, 30 cents per ½ pound.
4539. Genuine Java cinnamon (cassia), English, 10 cents per ¼ pound.
4547. Cinnamon, Java, ground in Washington.
4556. Ceylon cinnamon bark. Druggists.
4558. Ceylon cinnamon bark. Druggists, 15 cents per ounce.
4868. Cinnamon, pure, Baltimore, Md.
4869. Cinnamon, best, Baltimore, Md.

DENOMINATED CASSIA.

4557. Cassia bark. Druggists.
4559. Cassia bark, 10 cents per ounce. Druggists.
4640. Cassia bark, 10 cents per ounce. Druggists.
4906. Saigon cassia chips, collected in Baltimore markets by Z. D. Gilman.
4907. Cassia ligna bark, collected in Baltimore markets by Z. D. Gilman.
4908. Batavia bark, collected in Baltimore markets by Z. D. Gilman.
4909. Saigon cassia bark, collected in Baltimore markets by Z. D. Gilman.

A mechanical and microscopic examination showed that the ground specimens were hardly what they were represented to be.

4503, guaranteed pure cinnamon, proved to be a good quality of ground cassia.

4519 and 4529 consisted of substitution of cassia for cinnamon.

4531 was a low grade cassia mixed with considerable foreign material.

4539 was an excellent specimen of good cassia.

4547 was a poor quality cassia, adulterated with foreign material, but so finely ground as to make its identification impossible.

4868 and 4869, although labeled cinnamon, were mixtures of the lowest grade, consisting of cassia, turmeric ochre in small amount, mustard hulls or those of a similar seed, cracker dust, and burnt shells.

The remaining specimens were purchased unground, and, with the exception of the one English brand labeled Java cinnamon (cassia), no ground cassia could be purchased under such a designation.

The data obtained in a chemical way were as follows:

Analyses of cinnamon and cassia.

Serial No.	Description.	Sold as—	Consisting of—	Price per pound.	Water.	Ash.	Volatile oil.	Fixed oil, &c.	Crude fiber.	Albuminoids.	Undetermined.	Total.	Nitrogen.
				Cents.									
4502	Ceylon bark				10.00	3.70	3.14	3.30	16.18	3.80	59.88	100	.61
4556	do			*15	5.40	4.55	1.05	1.66	33.08	2.98	51.28	100	.48
4558	do			*15	7.93	3.40	.82	1.58	25.63	3.80	56.84	100	.62
4557	Cassia bark				9.42	2.35	.58	1.40	17.73	2.80	65.72	100	.45
4559	do			*15	9.01	1.75	.84	1.75	20.63	2.45	63.57	100	.39
4640	Buds			40	4.79	5.58	3.59	5.21	8.60	7.00	65.23	100	1.12
4906†	Cassia bark			10	9.49	8.23	1.01	2.13	26.29	4.20	48.65	100	.67
4907‡	do			09	11.04	2.48	1.21	1.86	15.45	2.63	65.33	100	.42
4908§	do			10	17.45	5.25	.55	.74	14.33	4.03	63.65	100	.64
4909‖	do			42	9.32	5.86	3.51	2.38	16.95	4.55	57.43	100	.73
4503	Commercial ground bark	Cinnamon	Cassia		5.60	4.80	3.70	3.92	29.88	2.00	56.10	100	.33
4519	do	do	do	60	9.37	3.36	.96	2.46	18.85	2.28	62.72	100	.36
4529	do	do	do	40	9.51	3.42	1.19	2.69	15.73	2.63	64.83	100	.42
4531	do	do	do	40	7.76	2.92	1.37	1.92	16.43	2.98	66.62	100	.48
4539	do	Cassia	Cassia	1.20	5.19	5.65	4.41	3.70	19.10	3.15	58.80	100	.50
4547	do	do	C. mixtures	40	6.45	2.50	1.00	1.96	18.43	2.10	63.56	100	.34
4868	do	do	do	16	8.73	4.02	1.90	2.86	16.88	4.55	61.06	100	.73
4869	do	do	do	14	11.21	3.74	1.15	2.13	19.18	4.20	58.39	100	.67

* Per ounce. † Saigon chips. ‡ Lignea bark. § Batavia bark. ‖ Saigon bark.

The preceding figures show that there is nothing particularly distinctive between the cinnamons and cassias, and that the determination of volatile oil points more to the character and value of the bark than any other, though, at the same time, there is nothing distinctive therein, as the variation in amount is so large in the cinnamons as to at times furnish samples containing far less volatile oil than even a fair cassia. The percentage of ash is extremely variable, depending on the age and quality of the bark. Saigon chips were found to have 8.23 per cent., while a specimen of unidentified cassia bark had only 1.75 per cent. Cinnamon bark will probably average less than cassia. Fiber, like ash, is extremely variable, and for the same reasons; 26.29 per cent. were found in Saigon chips and 33.08 per cent. in a cinnamon and from 14 to 20 in ordinary cassias. This determination, therefore, reveals nothing, and is of no assistance in detecting adulterants.

Albuminoids are variable, but within narrow limits, the extremes being 4.55 per cent. in Saigon bark and 2.45 in an unidentified cassia. The Batavia and Saigon barks appear to contain the most, over four per cent., and this percentage would seem to be an indication of *inferior quality.*

The amount of tannin in these barks is extremely small, not reaching in our specimens an equivalent of quercitannic acid by the Löwenthal process of two per cent. The addition, therefore, of material containing tannin can be readily detected, but in no case under our observation did such an addition occur.

Aside from the determination of volatile oil, chemical analysis seems to be of little value. The principal dependence must, with our present knowledge, be placed on the mechanical and microscopic examination, since the worst mixtures, 4868 and 4869, scarcely revealed in their composition the fact of their inferiority.

CLOVES.

Cloves are the flower-buds of an evergreen tree, *Eugenia caryophyllata*, growing wild in the Malaccas and introduced into Amboyna, the neighboring Zanzibar islands, Cayenne, and a few other places in the tropics. They are picked by hand when their development has reached a red color, and are dried in the sun, becoming dark brown. They are classed as East Indian, African, and American, and are valued in that order.

Flückiger and Hanbury's description of them is as follows:

> Cloves are about six-tenths of an inch in length, and consist of a long cylindrical calyx dividing above into four pointed spreading sepals which surround four petals, closely imbricated as a globular bud about two-tenths of an inch in diameter.
>
> The petals, which are of a lighter color than the rest of the drug and somewhat translucent from numerous oil cells, spring from the base of a four-sided epigynous disc, the angles of which are directed toward the lobes the calyx. The stamens, which are very numerous, are inserted at the base of the petals and are arched over the style. The latter, which is short and subulate, rises from a depression in the center of the disc. Immediately below it, and united with the upper portion of the calyx, is the

ovary, which is two-celled and contains many ovules. The lower end of the calyx (hypantium) has a compressed form; it is solid, but has its internal tissue far more porous than the walls. The whole calyx is of a deep rich brown, has a dull wrinkled surface, a dense fleshy texture, and abounds in essential oil, which exudes on simple pressure with the nail.

The varieties of cloves occurring in commerce do not exhibit any structural differences. Inferior kinds are distinguished by being less plump, less bright in tint, and less rich in essential oil. In London price-currents cloves are enumerated in the order of value thus: Penang, Bencoolen (Sumatra), Amboyna, Zanzibar.

A transverse section of the lower part of a clove shows a dark rhomboid zone, the tissue on either side of which is of a lighter hue. The outer layer beneath the epidermis exhibits a large number of oil cells, frequently as much as 300 micromillimeters in diameter. About 200 oil cells may be counted in one transverse section, so that the large amount of essential oil in the drug is well shown by its microscopic characters. The above-mentioned zone is chiefly made up of about 30 fibro-vascular bundles, another stronger bundle traversing the center of the clove. The fibro-vascular bundles, as well as the tissue bordering the oil cells, assume a greenish-black hue by alcoholic perchloride of iron. Oil cells are also largely distributed in the leaves, petals, and even stamens of *Eugenia*. No starch is found in it, however.

Preparations from whole cloves enable one to familiarize himself with the structure, which in the ground cloves is recognized with greater difficulty. In both cases the use of chloral hydrate solution is desirable, as the sections and fragments are otherwise not transparent. Preliminary examination of the powder in water for starch should, however, be made, as the starch granules swell in the chloral hydrate solution.

Among the fragments will be seen a large amount of débris of no apparent structure, but the larger pieces are chiefly the cells of the epidermis interspersed with the large oil cavities or cells, which are not as readily detected as in carefully prepared sections, being concealed by a layer of the epidermal cells. Next in prominence are the fibro-vascular bundles with their spiral vessels and with shreds of deep brown cellular matter attached. Pollen grains and, at times, whole anthers are present, and concretions of oxalate of lime. All these characteristic appearances are made out much more easily under polarized light, the long cells of the fibro-vascular bundles being optically active, as are also the pollen grains, oxalate of lime to a less degree, and the contents of the oil cells which are thus easily distinguished.

After a study of standard powder of cloves, the presence of adulterants is not difficult to recognize. Pimento is often added, and may be identified by the starch which it contains and the characters described under that spice.

Clove stems are said to be the commonest adulterant, and the presence in them of thick-walled stone cells and long yellow fibrous tissue serves as a means of recognition, since similar structures are not found in the clove, at least in the same abundance. The fruit of the clove is also added, and since it contains starch and a large embryo they are detected readily. We have not met either of these adulterants.

In the cheaper forms of ground cloves, where from the price it is evident that adulteration has been practiced, the common substitutes, which are added as diluents to all the spices, must be sought for and require no further experience beyond what has been obtained in the examination of pepper.

In most of the samples which were examined during the preparation of this report the microscope revealed nothing foreign, and for a decision as to their quality it was necessary to have recourse to chemical analysis. Two specimens from Baltimore of the same low grade as several of the spices previously mentioned were found, however, to be mixtures of all sorts of cheap material, containing mineral coloring matter, roasted shells or charcoal, corn, and hulls of seeds.

The sources of the cloves examined were as follows, being largely the ordinary article for sale at grocers:

SOURCES OF CLOVES.

4504. Whole cloves, guaranteed, Washington.
4903. Amboyna cloves, whole, direct from importers.
4904. Singapore cloves, whole, direct from importers.
4905. Clove stems, whole, direct from importers.
4641. Whole cloves, druggists', 10 cents per ounce.
4642. Whole cloves, druggists', 10 cents per ounce.
4643. Whole cloves, druggists', 10 cents per ounce.
4505. Ground cloves, guaranteed, Washington.
4520. Ground gloves, ground in Washington.
4540. Ground cloves, genuine Amboyna, 30 cents per ½ pound; English brand.
4548. Ground cloves, Washington grocers, second class, 26 cents per ½ pound.
4629. Ground cloves, Washington grocers, second class, 20 cents per ½ pound.
4630. Ground cloves, Washington grocers, 30 cents per pound.
4631. Ground cloves, "strictly pure," 40 cents per pound.
4632. Ground cloves, Washington grocers, second class, 30 cents per pound.
4633. Ground cloves, Washington grocers, second class, 30 cents per pound.
4873. Ground cloves, Baltimore Spice Mills; "pure."
4874. Ground cloves, Baltimore Spice Mills; "best."

The results of the analyses are as follows:

Analyses of cloves.

[Whole cloves.]

Serial No.	Source.	Weight of 100 cloves in grams.	Quality.	Water.	Ash.	Volatile oil.	Fixed oil and resin.	Crude fiber.	Albuminoids.	Nitrogen.	Oxygen equivalent.	As quercitannic acid.	Price per ¼ pound.
4504	Grocers			6.95	5.99	16.35	7.12	9.75	4.73	.76	4.06	15.86	
4641	Druggists			3.98	9.31	16.61	9.72	6.94	6.48	1.04	4.73	18.46	$0 40
4642	do			5.96	7.66	10.23	9.94	8.70	6.48	1.04	5.13	20.02	40
4643	do			2.90	13.05	15.87	10.07	8.55	7.00	1.12	3.00	11.70	40
4751	Extra quality			8.67	7.72	17.94	9.54	7.83	5.60	.90	5.43	21.19	
4903	Amboyna	10.505		8.78	5.25	18.89	10.24	6.18	5.42	.87	4.89	18.72	
4904	Singapore	8.710		10.67	5.50	13.52	9.95	9.08	5.42	.87	5.67	22.13	
4905	Stems			10.18	6.96	4.40	4.03	13.58	5.78	.92	5.96	23.24	

[Commercial ground cloves.]

Serial No.	Source.	Weight of 100 cloves in grams.	Quality.	Water.	Ash.	Volatile oil.	Fixed oil and resin.	Crude fiber.	Albuminoids.	Nitrogen.	Oxygen equivalent.	As quercitannic acid.	Price per ¼ pound.
4505			Fair	5.93	5.79	13.93	6.74	9.38	5.34	.86	5.60	21.84	
4520			Poor quality	8.86	5.98	9.88	6.88	11.75	4.20	.78	4.80	18.72	15
4540			do	8.30	5.85	12.40	6.58	11.10	4.38	.70	6.20	24.18	15
4548			do	8.79	6.05	10.21	7.44	10.93	4.38	.70	5.40	21.84	13
4629			do	8.78	8.48	8.45	5.36	10.75	5.95	.95	6.07	23.66	10
4630			do	9.58	10.73	6.52	4.94	12.30	5.78	.92	4.90	19.11	*30
4631			do	7.48	7.75	12.67	6.74	10.00	6.48	1.04	5.20	20.28	*40
4632			do	8.71	7.75	3.94	4.02	13.80	5.95	.95	2.89	11.28	15
4633			do	7.99	8.75	7.31	5.20	12.25	5.60	.90	5.33	21.58	10
4873			Adulterated	11.00	9.59	4.06	4.46	17.58	5.42	.87	3.00	11.70	*24
4874			do	8.62	8.35	3.59	4.47	17.78	5.08	.81	2.96	11.54	*21

* Per pound.

In comparison with these results it is of interest to refer to what has been already published.

Fluckiger states that from 16 to 20 per cent. of volatile oil is present, and gives details in regard to its composition and reactions; but beyond this nothing.

Dietsch gives the following figures as the percentages of oil to be expected in cloves from different sources:

	Per cent.
Amboyna	16 to 21
Zanzibar	12 to 17
Cayenne	9 to 12

There are one or two very old analyses in detail which are hardly reliable, and Dr. Ellis, of Toronto, has made investigations, still unpublished, in regard to the amount of tannin present, with a view to using the determination as a check on adulterants; but with these exceptions we are not aware of any other work upon the composition of this spice.

The authentic whole samples show that the percentage of water may be very variable, being at times as low as 2.90 per cent., and again as much as 10.67, which is high for so oily a substance. The ash, too, has rather wide extremes, varying from 5 to 13 per cent. The usual amount would not, however, be far from 5.50 to 6.50 per cent. Volatile oil falls in no case below the amount given by Dietsch, and serves as, perhaps, the best means of judging of the quality of the specimens. The extremes found in the pure specimens were 10.23 and 18.89 per cent., while but five out of the eleven ground specimens reached 10 per cent.

Other determinations do not seem especially characteristic. The extremes, which cannot be exceeded without casting suspicion, are:

	Per cent.
Water	11.00 to 2.75
Ash	13.00 to 5.00
Volatile oil	21.00 to 9.00
Fixed oil and resin	11.00 to 4.00
Crude fiber	10.00 to 6.00
Albuminoids	8.00 to 4.00

The determination of tannin, following the suggestions of Dr. Ellis, of Toronto, has been examined with results showing it to be of some value, but not as great as that of volatile oil. Our experience showed that it was as well to determine the matter oxidizable by permanganate, after removal of oil, &c., by Squibb's ether, as to make a more elaborate determination of tannin itself. In the best whole cloves from the importers the quercitannic acid equivalent of the oxidizable matter varied from 18.72 to 22.13 per cent., and in the stem reached 23.24 per cent. The determination will not, therefore, show the presence of stems in ground cloves. The amount fell also in one unidentified specimen of whole cloves to 11.70 per cent., but the quality of these buds was unknown. It is fair to assume, then, that good cloves should contain extractive oxidizable matter equivalent to 18 per cent. of quercitannic acid, or require an oxygen equivalent of about 4.50 per cent. for its reduction.

Of the 11 specimens of ground cloves examined, although none of them were of first quality, this tannin equivalent exceeded 18 in all but 3. The addition of stems and allspice would not be discovered, as both contain tannin in considerable amount. This determination is, then, in no way conclusive, but merely furnishes an indication which must be corroborated by other means. For the method of carrying it out reference must be made to our pages on analyses. As has been said, none of the ground specimens of cloves were first class, analyses showing that in only one case did the essential oil reach as high as 13.93 per cent., and although but 2 from Baltimore contained cheap foreign adulterants and none were sophisticated with allspice, all the specimens must have been made from a low grade of buds and many with the addition of large quantities of stems and spent cloves. The two cheapest specimens, 4873 and 4874, were, as has been said, terrible compounds of mineral coloring matter, leaving a dark ferruginous ash, corn meal, and hulls, evidence of which appears in the analyses from the low oil, 3.59 and 4.06 per cent., and the high crude fiber. The addition of so much organic matter low in ash conceals the presence of mineral coloring matter which is detected by its ferruginous appearance. In the ash of the whole buds, while there are at times some of a light reddish tinge, the color is distinctly or often dark green from the presence of manganese.

Our results show the universal and alarmingly poor quality of the commercial supply of ground cloves.

PIMENTO OR ALLSPICE.

Pimento is the fruit of *Pimenta officinalis*, an evergreen tree common in the West Indies. It is the only one of the common spices which had its origin in the New World. It is a small, dry, globular berry from two to three tenths of an inch in diameter, having a short style and surrounded by four short thick sepals which often, however, have become rubbed off, leaving a scar-like ring. The berry has a woody shell, or pericarp, easily cut, and of dark, ferruginous brown, rugose by means of minute tubercles filled with essential oil. It is two-celled, each cell containing a single seed. The seed is less aromatic than the pericarp.

Under the microscope the outer layer of the pericarp, just beneath the epidermis, appears as a collection of very large brown parenchymatous cells filled with oil. The more interior layers consist of thick walled or stone cells loaded with resin, the most characteristic structure of the pimento, parenchyma cells, and smaller crystals of calcic oxalate which are not easily seen. The whole tissue is traversed, but not plentifully, by fibro-vascular bundles. The seeds contain much starch in minute grains and have a few oil cells. The embryo is large and spirally curved. The hulls of the seeds consist of a delicate epidermis and of large thin-walled cells with light or dark red contents, which are very characteristic and are called by Hassall the port-wine cells, which should be examined in water, and after treatment with chloral hydrate, the starch grains being made out in the water preparation and the remaining structure among the particles rendered transparent by the chloral hydrate. Most prominent among the latter under polarized light, which is here a great assistance, are the stone cells or thick walled cells partly grouped and partly separate, and often, with plain light, showing shreds of parenchyma adherent to them. The brown cells which contain the oil are made out with less distinctness, but most striking are the red or port-wine cells of the seed hull, which are seen scattered everywhere, and in color and form are very characteristic. Shreds of the embryo are also now and then seen.

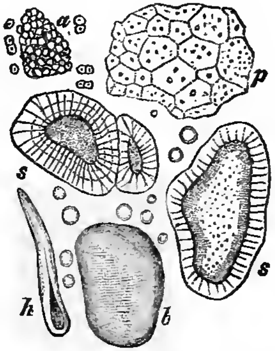

FIG. 13. Allspice powder. *a*, starch; *b*, port-wine cells; *h*, hairs; *s*, stone cells; *p*, parenchyma. ×240.

Schimper's diagramatic illustrations of this spice are here copied in Fig. 13, and serve as a slight aid to the recognition of the structures mentioned, but merely as suggestions, as nothing exactly corresponding to the drawings will be found in the ground powder.

Polarized light is a most important aid in examining this spice. It brings out strongly the stone-cells and ligneous tissue and differentiates therefrom the great mass of other matter. It also makes the oil cavities more distinct.

The adulteration of this spice does not often occur, owing to its cheapness. We have only found three cases, and those from Baltimore. In No. 4530 a substitution had been made for allspice, of which not a particle could be detected, of inferior cloves from which much of the volatile oil had been extracted, and Nos. 4877 and 487[illegible], where yellow corn and mineral coloring matter were plentiful. In these instances chemical analysis confirmed the microscopic examination.

Abroad clove stems are said to be largely used as an adulterant. They differ from cloves, as has been already explained, in the presence of numerous stone-cells and fibro-vascular bundles, and in Canada peas and roasted corn have been found. The presence of these cheap diluents should be sought for, as in peppers, but they are less common in this spice.

CHEMICAL COMPOSITION OF ALLSPICE.

But little has been published in regard to the proximate principles of the pimento. The amount of volatile oil is said by Flückiger to be from 3 to 4½ per cent., while starch and much tannin are present in the berry. Dragendorff has also found a minute quantity of an alkaloid of the odor of coumarine. Hassall gives analyses, which are too old to be of any value, and quotes Pereira as an authority for the fact that the essential oil of pimento is made up of two distinct oils, which, of course, may be true in one sense but is hardly of value as a definite distinction, both for this spice and cloves, as has been already remarked.

As a matter of fact but little is known of this spice. We have examined seven samples with the results here given:

4500. Whole allspice.
4501. Ground allspice, guaranteed pure, Washington.
4518. Ground allspice, ground in Washington.
4525. Ground allspice, ground in Baltimore.
4530. Ground allspice, ground in Baltimore.
4534. Ground allspice, ground in Baltimore.
4538. Ground allspice, English brand.
4877. Ground allspice, best, cheap grade, Baltimore.
4878. Ground allspice, pure, cheap grade, Baltimore.

All the samples were ground by different firms except the last two.

Analyses of pimento, allspice.

Serial No.	Description.	Water.	Ash.	Volatile oil.	Fixed oil.	Undetermined.	Crude fiber.	Albuminoids.	Nitrogen.	Tannin equivalent.	O required.
4500	Whole	6.19	4.01	5.15	6.15	59.28	14.83	4.38	.70	10.97	2.81
4501	Commercial ground	5.51	3.93	2.93	6.10	58.24	17.95	5.34	.86	13.10	3.36
4518	......do	8.03	4.83	2.07	5.50	57.20	18.00	4 38	.70	9.31	2.39
4525	......do	8.82	4.91	3.32	6.18	57.90	13.45	5.42	.87	9.39	2.40
4530	......do	11.59	6.02	8.17	7.64	59.57	11.93	5.08	.81	18.72	4.80
4534	......do	7.31	3.45	3.16	6.92	58.58	16.55	4.03	.61	12.74	3.27
4538	......do	8.71	4.50	1.29	5.35	55.90	18.83	5.42	.87	10.92	2.80
4877	......do	7.98	5.53	2.80	3.77	56.86	18.98	4.38	.70	8.27	2.12
4878	......do	7.31	5.19	1.82	1.60	56.45	23.60	4.03	.67	4.32	1.11

In our analyses there is considerable variation among the samples. Taking the whole berry as the standard the others fall off very much in quality, as judged by the volatile oil, with the exception of number 4530, which, too, from its low fiber, high ash, and high volatile and fixed oil is suspicious, and proves to be a substitution of cloves and clove stems. The microscope has shown that this sample is inferior cloves, and it must therefore be rejected.

The amount of essential oil in our best sample exceeds Flückiger's highest, but on the ground specimens falls off sadly, 4538 and 4878 being almost worthless as far as this valuable ingredient goes, perhaps in the first case having been exhausted for the preparation of the oil, although the ash is but little reduced in amount, and in the latter being nothing but mixtures, as shown under the microscope.

The other determinations show no important variation in the constituents, and estimation of the volatile oil would perhaps be the only thing necessary in a chemical way in examining it, without it is desired to go into the determination of tannin, which is as serviceable a means of discriminating among allspices as was found to be the case with cloves.

In the same way our results show that good allspice contains oxidizable matter extracted by water after removal of oil, etc., by ether equivalent to from 9–11.0 per cent. of quercetannic acid, the amount being considerably smaller than is found in cloves. This determination points out at once that specimens 4530 and 4878 are abnormal, and that 4534 is suspicious. It may be made of value and must be carried out in the same way as with cloves. Of the eight ground specimens examined three were adulterated and one was suspicious, so that even of this cheap spice we can hardly expect a pure supply without some protection.

NUTMEG.

Nutmegs are the interior kernel of the fruit of *Myristica fragrans*, a tree growing in various parts of the East, but principally in the Banda Islands.

Flückiger and Hanbury describe in a most excellent way their characteristics as follows:

> The fruit is a pendulous, globose drupe, about 2 inches in diameter, and not unlike a small round pear. It is marked by a furrow which passes around it, and by which at maturity its thick fleshy pericarp splits into two pieces, exhibiting in its interior a single seed, enveloped in a fleshy foliaceous mantle or arillus, of fine crimson hue, which is *mace*. The dark brown shining ovate seed is marked with impressions corresponding to the lobes of the arillus; and on one side, which is of paler hue and slightly flattened, a line indicating the raphe may be observed.
>
> The bony testa does not find its way into European commerce, the so-called nutmeg being merely the kernel or nucleus of the seed. Nutmegs exhibit nearly the form of their outer shell with a corresponding diminution in size. The London dealers esteem them in proportion to their size, the largest, which are about one inch long by eight-tenths of an inch broad, and four of which will weigh an ounce, fetching the highest price. If not dressed with lime, they are of a grayish brown, smooth yet coarsely furrowed and veined longitudinally, marked on the flatter side with a shallow groove. A transverse section shows that the inner seed coat (endopleura) penetrates into the albumen in long narrow brown strips, reaching the center of the seed, thereby imparting the peculiar marbled appearance familiar in a cut nutmeg. * * *
>
> The tissue of the seed can be cut with equal facility in any direction.

Of the microscopic structure they say:

> The testa consists mainly of long, thin, radially arranged rigid cells, which are closely interlaced and do not exhibit any distinct cavities. The endopleura which

forms the adhering coat of the kernel and penetrates into it, consists of soft walled, red brown tissue, with small scattered bundles of vessels. In the outer layers the endopleura exhibits small collapsed cells; but the tissue which fills the folds that dip into the interior consists of much larger cells

The tissue of the albumen is formed of soft-walled parenchymo, which is densely filled with conspicuous starch grains and with fat partly crystallized. Among the prismatic crystals of fat large, thick rhombic or six-sided tables may often be observed. With these are associated grains of albuminoid matter partly crystallized.

In carefully made preparations from the whole nutmeg the structure above described can be made out by care and patience. In the ground spice, however, of these characteristics only the interior parenchyma cells with their starchy contents can be seen when mounted in water, with the alternate use of common and polarized light. The fatty crystals we have not observed, and the fragments of the endopleura or red-brown tissue are characterized only by their.color. In chloral hydrate the starch cells and grains are swollen, but the red-brown tissue is much more transparent, sufficiently so, in fact, to reveal any differences between it and any adulterant which might bear a resemblance. There are but few bundles of fibers to be found. The structure as a whole is so simple that the addition of any foreign material can be readily detected.

CHEMICAL COMPOSITION OF NUTMEG.

Analysis of these samples of nutmegs of the following origin have been made:

4512. Whole limed nutmeg, grocers.
4513. Ground limed nutmeg, grocers.
4546. Ground in Baltimore.

Serial No.	Water.	Ash.	Volatile oil.	Fixed oil or fat.	Starch, &c.	Crude fiber.	Albuminoids.	Total.	Nitrogen.
4512	6.08	3.27	2.84	34.37	36.98	11.30	5.16	100.00	.83
4513	4.19	2.22	3.97	37.30	40.12	6.78	5.42	100.00	.87
4546	6.40	3.15	2.90	30.98	41.77	9.55	5.25	100.00	.84

Flückiger states that the volatile oil of nutmegs amounts to between 3 and 8 per cent., and that it is of the nature of a terpene. The fat or fixed oil he places at one-fourth of the weight of the nutmeg, or 25.0 per cent., the principal constituents being starch and albuminoids. Beyond these facts gathered by this authority nothing of value seems to have been published in regard to the proximate percentage composition of nutmegs.

Our results with only three samples are fairly concordant. The volatile oil hardly reached the limit which has been mentioned. It is no doubt a variable quantity. The fixed oil or fat, on the other hand, far exceeds what was expected. Our more perfect methods of extraction may be partly the cause of these results, and it may be also due to a resinification of the portion of the volatile oil, thus reducing the percentage of this substance and increasing that of the fat.

The amount of starch and allied substances seems fairly constant, while that of albuminoids is rather lower than would be expected. The fiber is also low, but our methods of determining so called fiber are so indefinite that such determinations are of only of comparative value.

Without any actual trials it would seem that determinations of oil and fat, and perhaps of albuminoids and fiber, would serve well to detect foreign substances which would certainly modify in a striking way the normal relations of these proximate principles.

At present nutmegs are almost entirely sold whole and grated in the kitchen. Attempts at adulteration have, therefore, been few in number.

MACE.

The mantle or arillus of the nutmeg, a coat between the outside pericarp and the seed, is known as mace when separated and dried. The coat is not continuous, but is a net-work the form of which is recognizable in the dried spice.

Of its microscopic structure Flückiger and Hanbury say:

> The uniform, small-celled angular parenchyme is interrupted by numerous brown oil cells of larger size. The inner part of the tissue contains also thin brown vascular bundles. The cells of the epidermis on either side are colorless, thick walled, longitudinally extended, and covered with a peculiar cuticle of broad, flat ribbon-like cells, which cannot, however, be removed as a continuous film. The parenchyme is loaded with small granules, to which a red color is imparted by Millon's test (solution of mercurous nitrate) and an orange hue by iodine. The granules consequently consist of albuminous matter, and starch is altogether wanting.

This statement has been fully confirmed, and the presence of starch claimed by several writers disproved. Iodine produces a peculiar deep brown color approaching the black-blue given with starch, which, in connection with the granules, has given rise to the statements that starch is present.

In the ground powder of mace all the details of structure described by Flückiger are readily made out, especially in chloral hydrate preparations with polarized light, as the brown vascular bundles, the ribbon-like and epidermal cells all are polarizing substances, while the large mass of granular parenchyma cells are not. The ribbon-like cells are particularly interesting in the varied forms they assume. Adulterations can be readily detected.

CHEMICAL COMPOSITION OF MACE.

Flückiger and Hanbury found in mace 8.2 per cent. of essential oil and 24.5 per cent. of aromatic balsam or resinified aromatic oil but no fat; also 1.4 per cent. of uncrystallizable sugar and 1.8 of mucilage, or a body between that and starch, blued by iodine. The composition of mace they therefore find to be very different from that of nutmeg, contrary to the assertion of Hassall.

Three samples, which we have examined, gave the following results:

4508. Whole mace, guaranteed.
4509. Ground mace, guaranteed.
4535. Ground mace, Baltimore.
4879. Ground mace, Baltimore, low grade.

Analyses of mace.

Serial number.	Water.	Ash.	Volatile oil.	Resin.	Undetermined.	Crude fiber.	Albuminoids.	Total.	Nitrogen.
4508	5.67	4.10	4.04	27.50	41.17	8.93	4.55	100.00	.73
4509	4.86	2.65	8.66	29.08	35.50	4.48	6.13	100.00	.98
4535	10.47	2.20	8.68	23.33	34.68	6.88	5.08	100.00	.81
4879	8.90	3.23	5.39	35.09	28.01	12.20	7.18	100.00	1.15

The percentages of volatile oil agree with the determinations of Flückiger except in the sample ground from the whole nutmeg in our laboratory, which is deficient. The amount probably should always reach 8 per cent. in good nutmegs. The resin found averages rather higher than in Flückiger's specimen, but the presence of so large an amount is certainly very characteristic of this spice, as well of the nutmeg itself, and the presence of adulterants would, without doubt, unless skillfully provided for, be detected by the absence of more than 20 per cent. of this extractive matter.

No qualitative estimation was made of sugar and gum; they were detected qualitatively in small amounts, but from the determinations of fiber and albuminoids it is evident that a large amount of extractive matter not starch is present, and as Flückiger found only about 3 per cent. of substances soluble in alcohol and water and no starch, and as the non-nitrogenous material removed by acids amounts to 35 per cent., it is probably one of the numerous members of the cellulose group, related to gum or starch, which is contained in the parenchyma cells. In the absence of adulterated specimens it is difficult to say what methods of procedure should be taken for their detection; but aside from the microscopical examination, which would readily reveal most adulterants, the chemical characteristics seem to be so marked as to make the presence of any foreign substance evident in the results of analysis.

APPENDIX A.

BIBLIOGRAPHY OF THE LITERATURE OF SPICES AND CONDIMENTS.

The following bibliography consists of a list of such books and papers of any interest as have appeared, to my knowledge, within the last fifteen years. There has seemed to be no necessity for including anything published earlier than 1870, as the progress of investigation has left everything behind which was done before then. Blyth, however, in his work entitled "Foods, their Composition and analyses," gives lists of the ancient literature of the subject, which can be referred to by those who are interested in the historic rather than the analytical aspects of the subject. The works which were published before the date named and which are of interest to the analyst may also be found in a list given by Dr. Battershall in his recent publication.

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APPENDIX B.

SOME OF THE LAWS RELATING TO ADULTERATION OF SPICES AND CONDIMENTS.

The following laws are the most valuable and important which have been recently enacted, and are of such character as to make their enforcement possible. There are a few other States having laws which are practically dead letters.

Some portions not relating to the subject of this part of the Bulletin are omitted.*

LEGISLATION.

The New York State general law, of 1881, for the prevention of the adulteration of food and drugs is as follows:

SECTION 1. No person shall, within this State, manufacture, have, offer for sale, or sell any article of food or drugs which is adulterated within the meaning of this act, and any person violating this provision shall be deemed guilty of a misdemeanor, and upon conviction thereof shall be punished by fine not exceeding fifty dollars for the first offense, and not exceeding one hundred dollars for each subsequent offense.

2. The term "food," as used in this act, shall include every article used for food or drink by man. The term "drug," as used in this act, shall include all medicines for internal and external use.

3. An article shall be deemed to be adulterated within the meaning of this act:

a. In the case of drugs.

(1) If, when sold under or by a name recognized in the United States Pharmacopœia, it differs from the standard of strength, quality, or purity laid down therein.

(2) If, when sold under or by a name not recognized in the United States Pharmacopœia, but which is found in some other pharmacopœia or other standard work on materia medica, it differs materially from the standard of strength, quality, or purity laid down in such work.

(3) If its strength or purity fall below the professed standard under which it is sold.

b. In the case of food or drink.

(1) If any substance or substances has or have been mixed with it so as to reduce or lower or injuriously affect its quality or strength.

(2) If any inferior or cheaper substance or substances have been substituted wholly or in part for the article.

(3) If any valuable constituent of the article has been wholly or in part abstracted.

(4) If it be an imitation of, or be sold under the name of, another article.

(5) If it consists wholly or in part of a diseased or decomposed, or putrid or rotten, animal or vegetable substance, whether manufactured or not, or, in te case of milk, if it is the produce of a diseased animal.

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* I am indebted to the secretaries of the various States and Dr. Battershall for the material here collected.

(6) If it be colored, or coated, or polished, or powdered, whereby damage is concealed, or it is made to appear better than it really is, or of greater value.

(7) If it contain any added poisonous ingredient, or any ingredient which may render such article injurious to the health of the person consuming it: *Provided,* That the State board of health may, with the approval of the governor, from time to time declare certain articles or preparations to be exempt from the provisions of this act: *And provided further,* That the provisions of this act shall not apply to mixtures or compounds recognized as ordinary articles of food, provided that the same are not injurious to health and that the articles are distinctly labeled as a mixture, stating the components of the mixture.

4. It shall be the duty of the State board of health to prepare and publish from time to time lists of the articles, mixtures, or compounds declared to be exempt from the provisions of this act in accordance with the preceding section. The State board of health shall also from time to time fix the limits of variability permissible in any article of food or drug or compound, the standard of which is not established by any national pharmacopœia.

5. The State board of health shall take cognizance of the interests of the public health as it relates to the sale of food and drugs and the adulteration of the same, and make all necessary investigations and inquiries relating thereto. It shall also have the supervision of the appointment of public analysts and chemists, and upon its recommendation whenever it shall deem any such officers incompetent the appointment of any and every such officer shall be revoked and be held to be void and of no effect. Within thirty days after the passage of this act the State board of health shall meet and adopt such measures as may seem necessary to facilitate the enforcement of this act, and prepare rules and regulations with regard to the proper methods of collecting and examining articles of food or drugs, and for the appointment of the necessary inspectors and analysts; and the State board of health shall be authorized to expend, in addition to all sums already appropriated for said board, an amount not exceeding ten thousand dollars for the purpose of carrying out the provisions of this act. And the sum of ten thousand dollars is hereby appropriated, out of any moneys in the treasury not otherwise appropriated, for the purposes in this section provided.

6. Every person selling or offering or exposing any article of food or drugs for sale, or delivering any article to purchasers, shall be bound to serve or supply any public analyst or other agent of the State or local board of health appointed under this act, who shall apply to him for that purpose, and on his tendering the value of the same, with a sample sufficient for the purpose of analysis of any article which is included in this act, and which is in the possession of the person selling, under a penalty not exceeding $50 for a first offense and $100 for a second and subsequent offense.

7. Any violation of the provisions of this act shall be treated and punished as a misdemeanor; and whoever shall impede, obstruct, hinder, or otherwise prevent any analyst, inspector, or prosecuting officer in the performance of his duty shall be guilty of a misdemeanor, and shall be liable to indictment and punishment therefor.

8. Any acts or parts of acts inconsistent with the provisions of this act are hereby repealed.

9. All the regulations and declarations of the State board of health made under this act from time to time, and promulgated, shall be printed in the statutes at large.

10. This act shall take effect at the expiration of ninety days after it shall become a law.

Amendment of April 29, 1885.

SECTION 1. The title of chapter 407 of the laws of 1881, entitled "An act to prevent the adulteration of food and drugs," is hereby amended to read as follows: "An act to prevent the adulteration of food, drugs, and spirituous, fermented, or malt liquors in the State of New York."

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3. Section 2 is hereby amended to read as follows:

"2. The term food as used in this act shall include every article of food or drink by man, including teas, coffees, and spirituous, fermented, and malt liquors. The term drug as used in this act shall include all medicines for internal or external use."

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5. Section 5 is hereby amended to read as follows:

"5. The State board of health shall take cognizance of the interests of the public health as relates to the sale of food, drugs, spirituous, fermented, and malt liquors, and the adulteration thereof, and make all necessary inquiries relating thereto. It shall have the supervision of the appointment of public analysts and chemists, and upon its recommendation, whenever it shall deem any such officers incompetent, the appointment of any and every such officer shall be revoked and be held to be void and of no effect. Within thirty days after the passage of this act, and from time to time thereafter as it may deem expedient, the said board of health shall meet and adopt such measures, not provided for by this act, as may seem necessary to facilitate the enforcement of this act, and for the purpose of making an examination or analysis of spirituous, fermented, or malt liquors sold or exposed for sale in any store or place of business not herein otherwise provided for, and prepare rules and regulations with regard to the proper methods of collecting and examining articles of food, drugs, spirituous, fermented, or malt liquors, and for the appointment of the necessary inspectors and analysts. The said board shall at least once in the calendar year cause samples to be procured, in public market or otherwise, of the spirituous, fermented, or malt liquors distilled, brewed, manufactured, or offered for sale in each and every brewery or distillery located in this State, and a test, sample, or analysis thereof to be made by a chemist or analyist duly appointed by said board of health. The samples shall be kept in vessels and in a condition necessary and adequate to obtain a proper test and analysis of the liquors contained therein. The vessels containing such samples shall be properly labeled and numbered by the secretary of said board of health, who shall also prepare and keep an accurate and proper list of the names of the distillers, brewers, or vendors, and opposite each name shall appear the number which is written or printed upon the label attached to the vessel containing the sample of the liquor manufactured, brewed, distilled, or sold. Such lists, numbers, and labels shall be exclusively for the information of the said board of health, and shall not be disclosed or published unless upon discovery of some deleterious substance prior to the completion of the analysis, except when required in evidence in a court of justice. The samples when listed and numbered shall be delivered to the chemist, analyst, or other officer of said board of health, and shall be designated and known to such chemist, analyst, or officer only by its number, and by no other mark or designation. The result of the analysis or investigation shall thereupon, and within a convenient time, be reported by the officer conducting the same to the secretary of said State board of health, setting forth explicitly the nature of any deleterious substance, compound, or adulteration which may be detrimental to public health and which has been found upon analysis in such samples, and stating the number of the samples in which said substance was found. Upon such examination or analysis the brewer, distiller, or vendor in whose sample of spirituous, fermented, or malt liquor such deleterious substances, compounds, or adulterations shall be found, shall be deemed to have violated the provisions of this act, and shall be punishable as prescribed in section 7 of this act."

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7. Section 7 of said chapter 407 of the laws of 1881 is hereby amended to read as follows:

"7. Upon discovering that any person has violated any of the provisions of this act, the State board of health shall immediately communicate the facts to the district attorney of the county in which the person accused of such violation resides or carries on business, and the said district attorney, upon receiving such communication or notification, shall forthwith commence proceeding for indictment and trial of the accused as prescribed by law in cases of misdemeanor."

8. The State board of health shall be authorized to expend, in addition to the sums already appropriated for said board, an amount not exceeding $3,000, for the purpose of carrying out the provisions of this act, in relation to spirituous, fermented, or malt liquors. And the sum of $3,000 is hereby appropriated out of any moneys in the treasury not otherwise appropriated and expended for the purposes of this act.

9. This act shall take effect immediately.

The New Jersey general law is the same as that of New York.

Following are the rules of the New Jersey board of health for its inspectors and analysts:

DUTIES OF INSPECTORS.

1. The inspector is to buy samples of food or drugs, and to seal each sample in the presence of a witness.
2. The inspector must affix to each sample a label bearing a number, his initials, and the date of purchase.
3. Under no circumstance is the inspector to inform the analyst as to the source of the sample before the analysis shall have been completed and formally reported to the president or secretary of the state board of health.
4. Inspectors are to keep a record of each sample as follows:
    (1) Number of sample.
    (2) Date and time of purchase.
    (3) Name of witness to sealing.
    (4) Name and address of seller.
    (5) Name and address of producer, manufacturer, or wholesaler, when known, with marks on original package.
    (6) Name of analyst and date of sending.
    (7) How sent to analyst.
5. If the seller desires a portion of the sample, the inspector is to deliver it under seal. The duplicate sample left with seller should have a label containing the same marks as are affixed to the portion taken by the inspector.
6. The inspector is to deliver the sample to the analyst, taking his receipt for the same, or he may send it by registered mail, express, or special messenger.

DUTIES OF THE ANALYSTS.

1. The analyst is to analyze the samples immediately upon receipt thereof.
2. Samples, with the exception of milk and similar perishable articles, are to be divided by the analyst and a portion sealed up, and a copy of the original label affixed. These duplicates are to be sent to the secretary of the state board of health at the end of each month, and to be retained by him until demanded for another analysis, as provided for in section 3 of these rules.
3. Should the result obtained by any analyst be disputed in any case, an appeal may be made to the State board of health, through its secretary, by the defendant or person selling the sample, or his attorney, and said secretary shall then require another member of the committee of public analysts to repeat the analysis, using the duplicate sample for such purpose. But when an appeal shall be made, a sum of money sufficient to cover the expenses of the second analysis shall be deposited with the president of the state board of health, which sum shall be paid over to the analyst designated by the president and secretary of the board to perform the second analysis, in case the analysis shall be found to agree with the first in all essential particulars.

4 In the case of all articles having a standard of purity fixed by any of the laws of the State, the certificate of the analyst should show the relation of the article in question to that standard.

5. Where standards of strength, purity, or quality are not fixed by law, the committee of analysts shall present to the State board of health such standard as in their judgment should be fixed.

6. Each analyst should keep a record book, in which should be entered notes, as follows:

(1) From whom the sample is received.
(2) Date, time, and manner in which the sample was received.
(3) Marks on package, sealed or not.
(4) Results of analysis in detail.

This record should be produced at each meeting of the committee.

7. At the completion of the analysis a certificate in the form given below should be forwarded to the person from whom the sample was received, and a duplicate copy sent to the State board of health.

CERTIFICATE.

To whom it may concern:

I, ——, a member of the committee of public analysts, appointed by the State board of health of New Jersey under provisions of an act entitled "An act to prevent the adulteration of food and drugs," approved March 25, 1881, do hereby certify that I received from ——, on the —— day of ——, 188–, a sample of ——, sealed as required by the rules of said board, and bearing the following words, to wit: ——

I carefully mixed said samples and have analyzed the same, and hereby certify and declare the results of my analyses to be as follows: ——.

[Signature.] —— ——.

EXCEPTIONS.

The following exceptions are adopted:

Mustard.—Compounds of mustard with rice flour, starch, or flour may be sold if each package is marked "Compound Mustard," and if not more than 25 per cent. of such substance is added to the mustard.

Coffee.—Compounds of coffee with chicory, rye, wheat, or other cereals, may be sold if the package is marked "A Mixture," and if the label states the per cent. of coffee contained in said mixture.

Oleomargarine and other imitation dairy products may be sold if each package is marked with the name of the substance, and in all respects fulfils the terms of the special law as to these.

Sirups.—When mixed with glucose, sirup may be sold if the package is marked "A Mixture."

The following are the statutes of the State of Massachusetts relating to the adulteration of food and drugs:

GENERAL LAWS RELATING TO ADULTERATION.

FOOD AND DRUGS.

SECTION 1. No person shall, within this Commonwealth, manufacture for sale, offer for sale, or sell any drug or article of food which is adulterated within the meaning of this act.

2. The term "drug" as used in this act shall include all medicines for internal or external use, antiseptics, disinfectants, and cosmetics. The term "food" as used herein shall include all articles used for food or drink by man.

3. An article shall be deemed to be adulterated within the meaning of this act—

(*a*) *In the case of drugs.*—(1) If, when sold under or by a name recognized in the United States Pharmacopœia, it differs from the standard of strength, quality, or purity laid down therein, unless the order calls for an article inferior to such standard, or unless such difference is made known or so appears to the purchaser at the time of such sale; (2) if, when sold under or by a name not recognized in the United States Pharmacopœia, but which is found in some other pharmacopœia, or other standard work on *materia medica*, it differs materially from the standard of strength, quality, or purity laid down in such work; (3) if its strength or purity falls below the professed standard under which it is sold.

(*b*) *In the case of food.*—(1) If any substance or substances have been mixed with it so as to reduce, or lower, or injuriously affect its quality or strength; (2) if any inferior or cheaper substance or substances have been substituted wholly or in part for it; (3) if any valuable constituent has been wholly or in part abstracted from it; (4) if it is an imitation of or is sold under the name of another article; (5) if it consists wholly or in part of a diseased, decomposed, putrid, or rotten animal or vegetable substance, whether manufactured or not, or in the case of milk, if it is the produce of a diseased animal; (6) if it is colored, coated, polished, or powdered, whereby damage is concealed, or if it is made to appear better or of greater value than it really is; (7) if it contains any added or poisonous ingredient, or any ingredient which may render it injurious to the health of a person consuming it.

4. The provisions of this act shall not apply to mixtures or compounds recognized as ordinary articles of food or drinks, provided that the same are not injurious to health and are distinctly labeled as mixtures or compounds. And no prosecutions shall at any time be maintained under the said act concerning any drug the standard of strength or purity whereof has been raised since the issue of the last edition of the United States Pharmacopœia, unless and until such change of standard has been published throughout the Commonwealth.

5. The State board of health, lunacy, and charity shall take cognizance of the interests of the public health relating to the sale of drugs and food and the adulteration of the same, and shall make all necessary investigations and inquiries in reference thereto, and for these purposes may appoint inspectors, analysts, and chemists, who shall be subject to its supervision and removal.

Within thirty days after the passage of this act the said board shall adopt such measures as it may deem necessary to facilitate the enforcement hereof, and shall prepare rules and regulations with regard to the proper methods of collecting and examining drugs and articles of food. Said board may expend annually an amount not exceeding ten thousand dollars for the purpose of carrying out the provisions of this act: *Provided, however,* That not less than three-fifths of the said amount shall be annually expended for the enforcement of the laws against the adulteration of milk and milk products.

6. Every person offering or exposing for sale, or delivering to a purchaser, any drug or article of food included in the provisions of this act, shall furnish to any analyst or other officer or agent appointed hereunder, who shall apply to him for the purpose and shall tender him the value of the same, a sample sufficient for the purpose of the analysis of any such drug or article of food which is in his possession.

7. Whoever hinders, obstructs, or in any way interferes with any inspector, analyst, or other officer appointed hereunder, in the performance of his duty, and whoever violates any of the provisions of this act, shall be punished by a fine not exceeding fifty dollars for the first offense, and not exceeding one hundred dollars for each subsequent offense.

8. The State board of health, lunacy, and charity shall report annually to the legislature the number of prosecutions made under said chapter, and an itemized account of all money expended in carrying out the provisions thereof.

9. An inspector appointed under the provisions of said chapter two hundred and sixty-three of the acts of the year eighteen hundred and eighty-two shall have the same powers and authority conferred upon a city or town inspector by section two of chapter fifty-seven of the public statutes.

10. Nothing contained in chapter two hundred and sixty-three of the acts of the year eighteen hundred and eighty-two shall be in any way construed as repealing or amending anything contained in chapter fifty-seven of the public statutes.

11. Before commencing the analysis of any sample, the person making the same shall reserve a portion which shall be sealed; and in case of a complaint against any person the reserved portion of the sample alleged to be adulterated shall upon application be delivered to the defendant or his attorney.

12. Whoever knowingly sells any kind of diseased, corrupted, or unwholesome provisions, whether for meat or drink, without making the same fully known to the buyer, shall be punished by imprisonment in the jail not exceeding six months, or by fine not exceeding two hundred dollars.

13. Whoever fraudulently adulterates, for the purpose of sale, bread or any other substance intended for food, with any substance injurious to health, or knowingly barters, gives away, sells, or has in possession with intent to sell, any substance intended for food, which has been adulterated with any substance injurious to health, shall be punished by imprisonment in the jail not exceeding one year, or by fine not exceeding three hundred dollars; and the articles so adulterated shall be forfeited and destroyed under the direction of the court.

14. Whoever adulterates, for the purpose of sale, any liquors used or intended for drink, with Indian cockle, vitriol, grains of paradise, opium, alum, capsicum, copperas, laurel-water, logwood, Brazil wood, cochineal, sugar of lead, or any other substance which is poisonous or injurious to health, and whoever knowingly sells any such liquor so adulterated, shall be punished by imprisonment in the State prison not exceeding three years; and the articles so adulterated shall be forfeited.

15. Whoever fraudulently adulterates, for the purpose of sale, any drug or medicine, or sells any fraudulently adulterated drug or medicine, knowing the same to be adulterated, shall be punished by imprisonment in the jail not exceeding one year or by fine not exceeding four hundred dollars; and such adulterated drugs and medicines shall be forfeited and destroyed under the direction of the court.

16. Whoever sells arsenic, strychnine, corrosive sublimate, or prussic acid, without the written prescription of a physician, shall keep a record of the date of such sale, the name of the article, the amount thereof sold, and the name of the person or persons to whom delivered; and for each neglect shall forfeit a sum not exceeding fifty dollars. Whoever purchases deadly poisons as aforesaid, and gives a false or fictitious name to the vendor, shall be punished by fine not exceeding fifty dollars.

CHAP. 171.—AN ACT concerning the adulteration of food and drugs.

Be it enacted, etc., as follows :

Section two of chapter two hundred and sixty-three of the acts of the year eighteen hundred and eighty-two is hereby amended so as to read as follows: The term "drug" as used in this act shall include all medicines for internal or external use, antiseptics, disinfectants, and cosmetics. The term "food" as used herein shall include confectionery, condiments, and all articles used for food or drink by man.—Approved April 29, 1886.

RULES AND REGULATIONS OF THE STATE BOARD OF HEALTH, LUNACY, AND CHARITY OF MASSACHUSETTS RELATIVE TO THE INSPECTION AND ANALYSIS OF FOOD AND DRUGS.

1. The state board of health, lunacy, and charity shall appoint analysts and inspectors, as provided in section 5 of chapter 263, acts of 1882.

2. It shall be the duty of the inspectors to procure samples of drugs and articles of food at such times and places as the health officer shall direct, in the manner provided in section 6 of chapter 263 of the acts of 1882, and in section 3 of chapter 289 of the acts of 1884, and in all acts amendatory of said provisions.

3. Under the direction of the health officer, one of the inspectors shall, for the identification of samples, affix a number to each sample of food or drugs obtained by him, beginning with number one, and taking every alternate or odd number thereafter, without limit; and the other inspector shall use and affix each alternate or even number, beginning with number two, and following such form of numbering, without limit, also, as far as may be directed. Under no circumstances shall an inspector convey any information to an analyst as to the source from which any sample was obtained.

4. The inspectors shall keep records of each sample, each record to include the following items:

(*a*) The inspector's number.

(*b*) The date of purchase or receipt of sample.

(*c*) The character of the sample.

(*d*) The name of the vender.

(*e*) The name of the city or town and street and number where the sample is obtained, and in the case of a licensed milk peddler, the number of his license.

(*f*) As far as possible, the names of manufacturers, producers, or wholesalers, with marks, brands, or labels stamped or printed upon goods.

5. It shall be the duty of the analysts so appointed to determine, under the direction of the health officer, by proper examination and analysis, whether articles of food and drugs, manufactured for sale, offered for sale, or sold within this Commonwealth, are adulterated within the meaning of chapter 263 of the acts and resolves passed by the general court of Massachusetts in 1882, and all acts amendatory thereof, adulteration being defined as follows, viz:

In the case of drugs, (1) If sold under or by a name recognized in the United States Pharmacopœia, it differs from the standard of strength, quality, or purity laid down therein, unless the order calls for an article inferior to such standard, or unless such difference is made known or so appears to the purchaser at the time of such sale. (2) If, when sold under or by a name not recognized in the United States Pharmacpœia, but which is found in some other pharmacopœia or standard work on materia medica, it differs materially from the standard of strength, quality, or purity laid down in such work. (3) If its strength or purity falls below the professed standard under which it is sold.

In the case of food, (1) If any substance or substances have been mixed with it, so as to reduce or lower or injuriously affect its quality or strength. (2) If any inferior or cheaper substance or substances have been substituted, wholly or in part, for it. (3) If any valuable constituent has been wholly or in part abstracted from it. (4) If it is an imitation of or is sold under the name of another article. (5) If it consists wholly or in part of a diseased, decomposed, putrid, or rotten animal or vegetable substance, whether manufactured or not, or in the case of milk, if it is the produce of a diseased animal. (6) If it is colored, coated, polished, or powdered, whereby damage is concealed, or if it is made to appear of better or of greater value than it really is. (7) If it contains any added poisonous ingredient, or any ingredient which may render it injurious to the health of the person consuming it.

6. It shall also be the duty of the analysts to receive such specimens of food and drugs for analysis as may be delivered to them by the health officer, or by the inspectors, and to examine the same. To avoid, as far as possible, all suggestion or danger of specimens having been tampered with, each analyst shall keep each specimen in his possession in a suitable and secure place, labeled in such a manner as to prevent any person from having access to the same, without the knowledge and presence of the analyst.

Analyses of perishable articles should be made promptly after they are received.

7. An analyst shall give no information, under any circumstances, regarding the result of any analysis to any person except to the health officer of the board, prior to any trial in court in reference to such analysis.

The analysts shall carefully avoid any error regarding the inspector's number attached to each sample, and shall report the results of their work in detail to the health officer.

In the case of all articles having a numerical standard provided by statute, the result of the analysis should show their relation to such standard.

8. Before beginning the analysis of any sample, the analyst shall reserve a portion, which shall be sealed, and in the event of finding the portion analyzed to be adulterated, he shall preserve the sealed portion, so that in case of a complaint against any person the last-named portion may, on application, be delivered by the health officer to the defendant or to his attorney.

9. Each analyst shall present to the health officer on the Thursday before the first Saturday of each month, a summary of the analyses made by him during the previous month.

Each analyst shall also present, on or before the first of January of each year, an annual report of the work done for the year ending on the 30th of September preceding.

10. The health officer shall have charge of the reports of analyses, and shall cause cases founded on such reports to be submitted to the courts for prosecution.

In each case of a retailer, and of every dealer not a manufacturer or producer, he may, if the party has not been previously complained of in court, issue a notice or warning of any violation of the law relative to the adulteration of food and drugs, and of the offender's liability to prosecution on a repetition of the sale.

11. Should the result obtained by any analyst be questioned in any given case, another analyst shall repeat the analysis, unless otherwise instructed by the board, provided a sufficient sum to meet the expense of the analysis be deposited with the health officer by any interested party feeling aggrieved, which sum will not be returned unless the second analysis fails to confirm the first in essential particulars.

12. Any appeal from the decision of an analyst shall be filed with the health officer, who shall report it, and any matter in controversy, to the board, giving his judgment thereon, and the board shall supervise and control the action of its officers, in executing the law.

13. Where standards of strength, quality, or purity are not fixed by the act, the analysts shall present to the health officer such standard as in their judgment should be fixed, and the health officer shall report the same to the board for its action. The standards set by the British Society of Public Analysts will be followed as nearly as practicable, until otherwise ordered.

14. Whenever a drug or preparation not described in a National Pharmacopœia or other standard work on materia medica, shall be manufactured, offered for sale, or used in this State, the standard of such drug, and the standard and proportion of the ingredients of such preparation, and the range of variability from such standard or standards shall be ascertained by the analysts, who shall report the same through the health officer to the board.

15. The analysts shall occupy such time in the performance of their respective duties as a reasonable compliance with the terms of the statute shall require, and shall be present one hour of each day at such time of the day and at such place as shall be designated by the committee on health of the board, to meet the convenience of interested parties and the public.

16. The compensation of the analyst of articles of food shall be at the rate of $1,500 per annum, and that of the analyst of drugs shall be at the rate of $1,000.

That of the analyst of milk for the ten eastern counties of the Commonwealth shall be at the rate of $800 per annum, and that of the analyst of the four western counties shall be at the rate of $500 per annum.

The compensation of each inspector shall be at the rate of $1,000 per annum.

The laws of Michigan are as follows:

ADULTERATION OF FOODS, DRINKS, DRUGS, OR MEDICINES.

208. (7727.) Sec. 2. If any person shall fraudulently adulterate, for the purpose of sale, any substance intended for food, or any wine, spirits, malt liquor, or other liquor intended for drinking, he shall be punished by imprisonment in the county jail not more than one year, or by fine not exceeding three hundred dollars, and the article so adulterated shall be forfeited and destroyed.—§9317.

209. (7728.) Sec. 3. If any person shall fraudulently adulterate, for the purpose of sale, any drug or medicine, in such manner as to render the same injurious to health, he shall be punished by imprisonment in the county jail not more than one year, or by fine not exceeding four hundred dollars, and such adulterated drugs and medicines shall be forfeited and destroyed.—§9318.

ADULTERATION OF FOODS, DRINKS, AND MEDICINES. AND SALE THEREOF WHEN ADULTERATED.

Act No. 254, laws of 1881, entitled "An Act to prevent and punish the adulteration of articles of food, drink, and medicine, and the sale thereof when adulterated."

210. Section 1. *The People of the State of Michigan enact,* That no person shall mix, color, stain, or powder, or order or permit any other person to mix, color, stain, or powder any article of food with any ingredient or material so as to render the article injurious to health, with the intent that the same may be sold; and no person shall knowingly sell or offer for sale any such article so mixed, colored, stained, or powdered.—§9324.

211. Sec. 2. No person shall, except for the purpose of compounding in the necessary preparation of medicine, mix, color, stain, or powder, or order or permit any other person to mix, color, stain, or powder, any drug or medicine with any ingredient or ingredients or materials so as to affect injuriously the quality or potency of such drug or medicine, with intent to sell the same, or shall sell or offer for sale any such drug or medicine so mixed, colored, stained, or powdered.—§9325.

212. Sec. 3. No person shall mix, color, stain, or powder any article of food, drink, or medicine, or any article which enters into the composition of food, drink, or medicine, with any other ingredient or material, whether injurious to health or not, for the purpose of gain or profit, or sell or offer the same for sale, or order or permit any other person to sell or offer for sale any article so mixed, colored, stained, and powdered, unless the same be so manufactured, used, or sold, or offered for sale under its true and appropriate name, and notice that the same is mixed or impure is marked, printed, or stamped upon each package, roll, parcel, or vessel containing the same, so as to be and remain at all times readily visible, or unless the person purchasing the same is fully informed by the seller of the true name and ingredients (if other than such as are known by the common name thereof) of such article of food, drink, or medicine at the time of making sale thereof or offering to sell the same.—§9326.

213. Sec. 4. No person shall mix any glucose or grape sugar with syrup, honey, or sugar, intended for human food, or any oleomargarine, suine, beef fat, lard, or any other foreign substance, with any butter or cheese intended for human food, or shall mix or mingle any glucose or grape sugar or oleomargarine with any article of food, without distinctly marking, stamping, or labeling the article, or the package containing the same, with the true and appropriate name of such article, and the percentage in which glucose or grape sugar, oleomargarine, or suine, enter into its composition; nor shall any person sell, or offer for sale, or order or permit to be sold, or offered for sale, any such food into the composition of which glucose, or grape sugar, or oleomargarine, or suine has entered, without at the same time informing the buyer of the fact, and the proportions in which such glucose or grape sugar, oleomargarine, or suine has entered into its composition.—§9327.

214. Sec. 5. Any person convicted of violating any provision of any of the foregoing sections of this act shall be fined not more than fifty dollars or imprisoned in the county jail not exceeding three months.—§9328.

215. Sec. 6. It is hereby made the duty of the prosecuting attorneys of this State to appear for the people and to attend to the prosecution of all complaints under this act in all the courts in their respective counties.—§9329.

216. Sec. 7. All acts and parts of acts inconsistent with the provisions of this act are hereby repealed.—§9330.

The Canadian law is as follows, in addition to which there is an elaborate act respecting the inspection of staple articles of Canadian produce:

Chapter 107.—An Act respecting the adulteration of food, drugs, and agricultural fertilizers.

Her Majesty, by and with the advice and consent of the Senate and House of Commons of Canada, enacts as follows:

SHORT TITLE.

1. This act may be cited as "*The adulteration act.*"—48-49 V., c. 67, s. 1.

INTERPRETATION.

2. In this act, unless the context otherwise requires—

(*a*) The expression "food" includes every article used for food or drink by man or by cattle.

(*b*) The expression "drug" includes all medicines for internal or external use for man or for cattle.

(*c*) The expression "agricultural fertilizer" means and includes every substance imported, manufactured, prepared, or disposed of for fertilizing or manuring purposes which is sold at more than ten dollars per ton and which contains phosphoric acid or ammonia or its equivalent of nitrogen.

(*d*) The expression "officer" means any officer of inland revenue or any person authorized under this act or "*the fertilizers act*" to procure samples of articles of food, drugs, or agricultural fertilizers and to submit them for analysis.

(*e*) Food shall be deemed to be "adulterated" within the meaning of this act—

(1) If any substance has been mixed with it so as to reduce or lower or injuriously affect its quality or strength.

(2) If any inferior or cheaper substance has been substituted, wholly or in part, for the article.

(3) If any valuable constituent of the article has been wholly or in part abstracted.

(4) If it is an imitation of, or is sold under the name of, another article.

(5) If it consists wholly or in part of a diseased or decomposed or putrid or rotten animal or vegetable substance, whether manufactured or not, or, in the case of milk or butter, if it is the produce of a diseased animal or of an animal fed upon unwholesome food.

(6) If it contains any added poisonous ingredient or any ingredient which may render such an article injurious to the health of a person consuming it.

(*f*) Every drug shall be deemed to be "adulterated" within the meaning of this act—

(1) If, when sold or offered or exposed for sale under or by a name recognized in the British or United States pharmacopœia, it differs from the standard of strength, quality, or purity laid down therein.

(2) If, when sold or offered or exposed for sale under or by a name not recognized in the British or United States pharmacopœia, but which is found in some other generally recognized pharmacopœia or other standard work on *materia medica*, it differs from the standard of strength, quality, or purity laid down in such work.

(3) If its strength or purity falls below the professed standard under which it is sold or offered or exposed for sale.

(*g*) Provided, that the foregoing definitions as to the adulteration of food and drugs shall not apply—

(1) If any matter or ingredient not injurious to health has been added to the food or drug because the same is required for the production or preparation thereof as an article of commerce, in a state fit for carriage or consumption, and not fraudulently to increase the bulk, weight, or measure of the food or drug or to conceal the inferior quality thereof, if such articles are distinctly labeled as a mixture, in conspicuous characters, forming an inseparable part of the general label, which shall also bear the name and address of the manufacturer.

(2) If the food or drug is a proprietary medicine, or is the subject of a patent in force, and is supplied in the state required by the specification of the patent.

(3) If the food or drug is unavoidably mixed with some extraneous matter in the process of collection or preparation.

(4) If any articles of food not injurious to the health of the person consuming the same are mixed together and sold or offered for sale as a compound, and if such articles are distinctly labeled as a mixture, in conspicuous characters, forming an inseparable part of the general label, which shall also bear the name and address of the manufacturer.

(*h*) Every agricultural fertilizer shall be deemed to be "adulterated" within the meaning of this act if, when sold, offered, or exposed for sale, the chemical analysis thereof shows a deficiency of more than one per cent. of any of the chemical substances, the percentages whereof are required to be specified in the certificate, by "*the fertilizers act*" required to be affixed to each barrel, box, sack, or package containing the same, or (if the agricultural fertilizer is in bulk) to be produced to the inspector; or if it contains less than the minimum percentage of such substances required by the said act to be contained in such fertilizer. 48–49 V., c. 67, s. 2.

ANALYSIS.

3. The governor in council may appoint one or more persons possessing competent medical, chemical, and microscopical knowledge as analysts of food, drugs, and agricultural fertilizers purchased, sold, or exposed or offered for sale within such territorial limits as are assigned to each of them respectively, and may also select from among the aforesaid analysts so appointed, or may appoint, in addition thereto, a chief analyst, who shall be attached to the staff of the department of inland revenue at Ottawa.

(2) No analyst shall be appointed until he has undergone an examination before a special examining board appointed by the governor in council, and until he has obtained from such board a certificate setting forth that he is duly qualified to perform the duties attached to the office of analyst. 48–49 V., c. 67, s. 3; 49 V., c. 41, s. 1.

4. The governor in council may cause such remuneration to be paid to such chief analyst and to such analysts as he deems proper, and such remuneration, whether by fees, or salary, or partly in one way, and partly in the other, may be paid to them out of any sums voted by Parliament for the purposes of this act. 48–49 V., c. 67, s. 4.

5. The officers of inland revenue, the inspectors and deputy inspectors of weights and measures, and the inspectors and deputy inspectors acting under "*the general inspection act*," or any of them, shall, when required so to do by any regulation made in that behalf by the minister of inland revenue, procure and submit samples of food, drugs, or agricultural fertilizers suspected to be adulterated, to be analyzed by the analysts appointed under this act. 48–49 V., c. 67, s. 5.

6. The council of any city, town, county, or village may appoint one or more inspectors of food, drugs, and agricultural fertilizers; and such inspectors shall, for the purposes of this act, have all the powers by this act vested in officers of inland revenue; and any such inspector may require any public analyst to analyze any sam-

ples of food, drugs, or agricultural fertilizers collected by him, if such samples have been collected in accordance with the requirements of this act.

(2) The said analyst shall, upon tender of the fees fixed for the analysis of such class of articles by the governor in council, forthwith analyze the same, and give the inspector a certificate of such analysis.

(3) Such inspector may prosecute any person manufacturing, selling, or offering or exposing for sale within the city, county, town, or village for which he is appointed inspector, any article of food, drug, or agricultural fertilizer which has been certified by any public analyst to have been adulterated within the meaning of this act.

(4) Notwithstanding any other provision of this act in respect of the disposition of penalties, all penalties imposed and recovered at the suit of any such inspector shall be paid into the revenue of the city, county, town, or village by the council of which such inspector was appointed, and may be distributed in such manner as the council of such city, county, town, or village by by-law directs. 48–49 V., c. 67, s. 6.

7. Any officer may procure samples of food, drugs, or agricultural fertilizers which have not been declared exempt from the provisions of this act, from any person who has such articles in his possession for the purpose of sale, or who sells or exposes the same for sale; and he may procure such samples either by purchasing the same or by requiring the person in whose possession they are to show him and allow him to inspect all such articles in his possession, and the place or places in which such articles are stored, and to give him samples of such articles, on payment or tender of the value of such samples. 48–49 V., c. 67, s. 7.

8. If the person who has such articles in his possession, or his agent or servant, refuses or fails to admit the officer, or refuses or omits to show all or any of the said articles in his possession, or the place in which any such articles are stored, or to permit the officers to inspect the same, or to give any samples thereof, or to furnish the officer with such light or assistance as he requires, when required so to do in pursuance of this act, he shall be liable to the same penalty as if he knowingly sold or exposed for sale adulterated articles knowing them to be adulterated. 48–49 V., c. 67, s. 8.

9. The officer purchasing any article with the intention of submitting the same to be analyzed, shall, after the purchase has been completed, forthwith notify the seller or his agent selling the article of his intention to have the same analyzed by the public analyst, and shall, except in specific cases, respecting which provision is made by the governor in council, divide the article into three parts—to be then and there separated, and each part to be marked and sealed up, as its nature permits—and shall deliver one of the parts to the seller or his agent, if required by him so to do.

(2) He shall transmit another of such parts to the minister of inland revenue for submission to the chief anaylist in case of appeal, and shall submit the remaining part to the analyst for the district within which the samples were taken, unless otherwise directed by the minister of inland revenue. 48–49 V., c. 67, s. 9.

10. The person from whom any sample is obtained under this act may require the officer obtaining it to annex to the vessel or package containing the part of the sample which he is hereby required to transmit to the minister of inland revenue the name and address of such person, and to secure, with a seal or seals belonging to him, the vessel or package containing such part of the sample, and the address annexed thereto, in such manner that the vessel or package cannot be opened, or the name and address taken off, without breaking such seals; and the certificate of the chief analyst shall state the name and address of the person from whom the said sample was obtained, that the vessel or package was not open, and that the seals, securing to the vessel or package the name and address of such person, were not broken until such time as he opened the vessel or package for the purpose of making his analysis; and in such case no certificate shall be receivable in evidence, unless there is contained therein such statement as above, or a statement to the like effect. 48–49 V., c. 67, s. 10.

11. When the officer has, by either of the means aforesaid, procured samples of the articles to be analyzed, he shall cause the same to be analyzed by one of the analysts appointed under this act, and if it appears to the analyst that the sample is adulterated within the meaning of this act, he shall certify such fact, stating in such certificate, in the case of an article of food or a drug, whether such adulteration is of a nature injurious to the health of the person consuming the same; and the certificate so given shall be received as evidence in any proceedings taken against any person in pursuance of this act, subject to the right of any person against whom proceedings are taken to require the attendance of the analyst, for the purpose of cross-examination. 48–49 V., c. 67, s. 11.

12. If the vendor of the article respecting which such certificate is given deems himself aggrieved thereby, he may, within forty-eight hours of the receipt of the first notification of the intention of the officer or other purchaser to take proceedings against him (whether such notification is given by the purchaser or by the ordinary process of law), notify the said officer or purchaser in writing that he intends to appeal from the decision of the analyst to the judgment of the chief analyst: and in such case the officer or purchaser shall transmit such notification to the chief analyst, and the chief analyst shall, with all convenient speed, analyze the part of the sample transmitted to the minister of inland revenue for that purpose, and shall report thereon to the said minister; and the decision of such chief analyst shall be final, and his certificate thereof shall have the same effect as the certificate of the analyst in the next preceding section mentioned. 48–49 V., c. 67, s. 12.

13. Every analyst appointed under this act shall report quarterly to the minister of inland revenue the number of articles of food, drugs, and agricultural fertilizers analyzed by him under this act during the preceding quarter, and shall specify the nature and kind of adulterations detected in such articles of food, drugs, and agricultural fertilizers; and all such reports, or a synopsis of them, and the names of the vendors or persons from whom obtained, and of the manufacturers when known, shall be printed and laid before Parliament as an appendix to the annual report of the said minister. 48–49 V., c. 67, s. 13.

ADULTERATION.

14. No person shall manufacture, expose or offer for sale, or sell any food, drug, or agricultural fertilizer which is adulterated within the meaning of this act. 48–49 V., c. 67, s. 14.

15. If milk is sold, or offered or exposed for sale, after any valuable constituent of the article has been abstracted therefrom, or if water has been added thereto, or if it is the product of a diseased animal fed upon unwholesome food, it shall be deemed to have been adulterated in a manner injurious to health, and such sale, offer, or exposure for sale shall render the vendor liable to the penalty hereinafter provided in respect to the sale of adulterated food; except that skimmed milk may be sold as such if contained in cans bearing upon their exterior, within twelve inches of the tops of such vessels, the word "skimmed" in letters of not less than two inches in length, and served in measures also similarly marked; but any person supplying such skimmed milk, unless such quality of milk has been asked for by the purchaser, shall not be entitled to plead the provisions of this section as a defense to or in extenuation of any violation of this act:

(2) Nothing in this section shall be interpreted to permit or warrant the admixture of water with milk, or any other process than the removal of cream by skimming. 48–49 V., c. 67, s. 15.

16. Vinegar sold, or offered or exposed for sale, shall be deemed to be adulterated in a manner injurious to health if any mineral acid has been added thereto, or if it contains any soluble salt having copper or lead as a base thereof—whether such mineral acid or salt is added either during the process of manufacture or subsequently. 48–49 V., c. 67, s. 16.

17. Alcoholic, fermented, or other potable liquors sold, or offered or exposed for sale, shall be deemed to have been adulterated in a manner injurious to health if they are found to contain any of the articles mentioned in the schedule of this act, or any article hereafter added to such schedule by the governor in council. 48–49 V., c. 67, s. 17.

18. The governor in council may from time to time declare certain articles or preparations exempt in whole or in part from the provisions of this act, and may add to the schedule to this act any article or ingredient the addition of which is by him deemed necessary in the public interest; and every order in council in that behalf shall be published in the Canada Gazette, and shall take effect at the expiration of thirty days from the date of such publication. (48–49 V., c. 67 s 18.)

19. The governor in council shall from time to time cause to be prepared and published lists of the articles, mixtures, or compounds declared exempt from the provisions of this act in accordance with the next preceding section, and shall also from time to time fix the limits of variability permissible in any article of food or drug or compound the standard of which is not established by any such pharmacopœia or standard work as is hereinbefore mentioned; and the orders in council fixing the same shall be published in the Canada Gazette, and shall take effect at the expiration of thirty days after the publication thereof. (48–49 V., c. 67, s. 19.)

20. Whenever any article of food, any drug, or any agricultural fertilizer is reported by any analyst as being adulterated within the meaning of this act, the minister of inland revenue may, if he thinks fit, order such article, and all other articles of the same kind and quality which were in the same place at the time the article analyzed was obtained, to be seized by any officer of customs or inland revenue, and detained by him until an analysis of samples of the whole is made by the chief analyst. (48, 49 V., c. 67, s. 20.)

21. If the chief analyst reports to the minister of inland revenue that the whole or any part of such articles are adulterated, the minister may declare such articles, or so much thereof as the chief analyst reports as being adulterated, to be forfeited to the Crown; and such articles shall thereupon be disposed of as the minister directs. (48–49 V., c. 67, s. 21.

PENALTIES.

22. Every person who willfully adulterates any article of food or any drug, or orders any other person so to do, shall—

(*a*) If such adulteration is within the meaning of this act deemed to be injurious to health, for the first offense incur a penalty not exceeding fifty dollars and not less than ten dollars and costs, and for each subsequent offense a penalty not exceeding two hundred dollars and not less than fifty dollars and costs.

(*b*) If such adulteration is within the meaning of this act deemed not to be injurious to health, incur a penalty not exceeding thirty dollars and costs, and for each subsequent offense a penalty not exceeding one hundred dollars and not less than fifty dollars and costs. (49–49 V., c. 67, s. 22.)

23. Every person who, by himself or his agent, sells, offers for sale, or exposes for sale any article of food or any drug which is adulterated within the meaning of this act shall—

(*a*) If such adulteration is within the meaning of this act deemed to be injurious to health, for a first offense incur a penalty not exceeding fifty dollars and costs, and for each subsequent offense a penalty not exceeding two hundred dollars and not less than fifty dollars and costs.

(*b*) If such adulteration is within the meaning of this act deemed not to be injurious to health, incur for each such offsense a penalty not exceeding fifty dollars and not less than five dollars and costs.

(2) *Provided,* That if the person accused proves to the court before which the case is tried that he did not know of the article being adulterated, and shows that he could not, with reasonable diligence, have obtained that knowledge, he shall be sub-

ject only to the liability to forfeiture under the twenty-first section of this act. (48–49 V., c. 67, s. 23.)

24. Every compounder or dealer in and every manufacturer of intoxicating liquors who has in his possession or in any part of the premises occupied by him as such any adulterated liquor, knowing it to be adulterated, or any deleterious ingredient specified in the schedule hereto, or added to such schedule by the governor in council, for the possession of which he is unable to account to the satisfaction of the court before which the case is tried, shall be deemed knowingly to have exposed for sale adulterated food, and shall incur for the first offense a penalty not exceeding one hundred dollars, and for each subsequent offense a penalty not exceeding four hundred dollars. (48–49 V., c. 67, s. 24.)

25. Every person who knowingly attaches to any article of food or any drug any label which falsely describes the article sold or offered or exposed for sale, shall incur a penalty not exceeding one hundred dollars and not less than twenty dollars and costs. (48-49 V., c. 67, s. 25.)

26. Every penalty imposed and recovered under this act shall, except as herein otherwise provided, and except in the case of any suit, action, or prosecution brought or instituted under the provisions of the next following section, be paid over to the minister of finance and receiver-general, and shall form part of the consolidated revenue fund. (48–49 V., c. 67, s. 26.)

GENERAL PROVISIONS.

27. Nothing herein contained shall be held to preclude any person from submitting any sample of food, drug, or agricultural fertilizer for analysis to any public analyst, or from prosecuting the vendor thereof, if such article is found to be adulterated, but the burden of proof of sale, and of the fact that the sample was not tampered with after purchase, shall be upon the person so submitting the same:

(2) Any public analyst shall analyze such sample on payment of the fee prescribed in respect of such article or class of article by the governor in council. (48–49 V., c. 67, s. 27.)

28. Any expenses incurred in analyzing any food, drug, or agricultural fertilizer, in pursuance of this act, shall, if the person from whom the sample is taken is convicted of having in his possession, selling, offering or exposing for sale, adulterated food, drugs, or agricultural fertilizers, in violation of this act, be deemed to be a portion of the cost of the proceedings against him, and shall be paid by him accordingly; and in all other cases such expenses shall be paid as part of the expenses of the officer, or by the person who procured the sample, as the case may be. (48–49 V., c. 67, s. 28.)

29. The governor in council may, from time to time, make such regulations as to him seem necessary, for carrying the provisions of this act into effect. (48–49 V., c. 67, s. 20.)

30. The provisions of "the inland revenue act," whether enacted with special reference to any particular business or trade, or with general reference to the collection of the revenue, or the prevention, detection, or punishment of fraud or neglect in relation thereto, shall extend, apply, and be construed, and shall have effect with reference to this act, as if they had been enacted with special reference to the matters and things herein provided for.

(2) Every penalty imposed under this act may be enforced and dealt with as if imposed under the said act, and every compounder, and the apparatus used by him, and the place in which his business is carried on, and the articles made or compounded by him, or used in compounding any such article, shall be "subject to excise" under the said act. (48–49 V., c. 67, s. 30.)

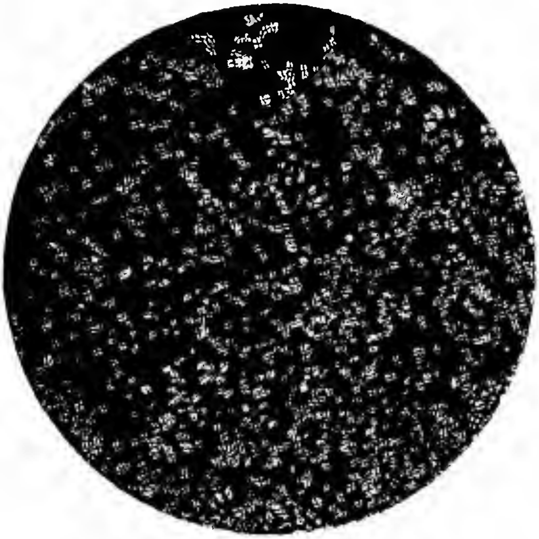
SCHEDULE.

Cocculus indicus, chloride of sodium (otherwise common salt), copperas, opium, cayenne pepper, picric acid, Indian hemp, strychnine, tobacco, darnel seed, extract of logwood, salts of zinc, copper, or lead, alum, methyl alcohol and its derivatives, amyl alcohol, and any extract or compound of any of the above ingredients.

The English laws will be found in Hassall or Blyth.

INDEX

Fig. 27.



MARUNTA STARCH

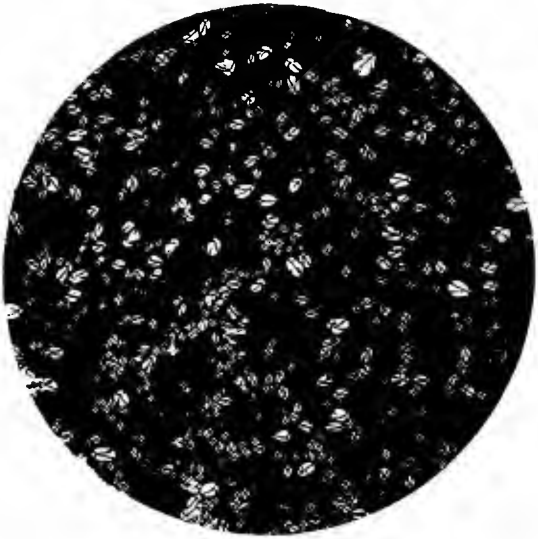
Fig. 28.

MARUNTA STARCH X 140

PHOTO BY CLIFFORD RICHARDSON.

A Hoen & Co. Lithocaustic

Fig. 29.



POTATO STARCH X 43

Fig. 30.

POTATO STARCH

PHOTO BY CLIFFORD RICHARDSON.

A. Hoen & Co., Lithocaustic

Fig. 31.

POTATO STARCH x 144

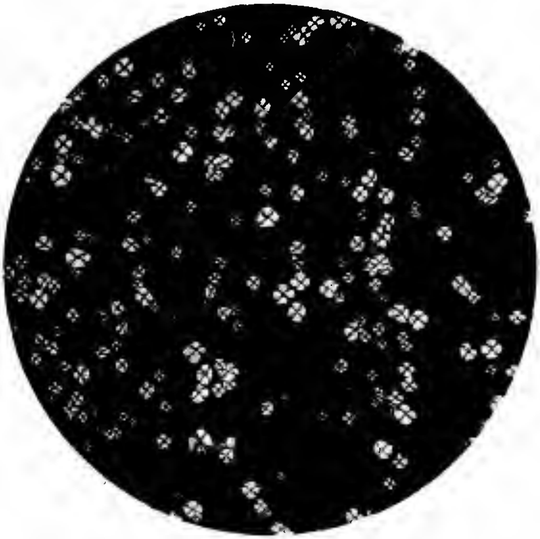
Fig. 32.

MARUNTA STARCH x 144

PHOTO BY CLIFFORD RICHARDSON.

A Hoen & Co., Lithocaustic

Fig. 33.



MAIZE STARCH X 145

Fig. 34.

WHEAT STARCH X 145

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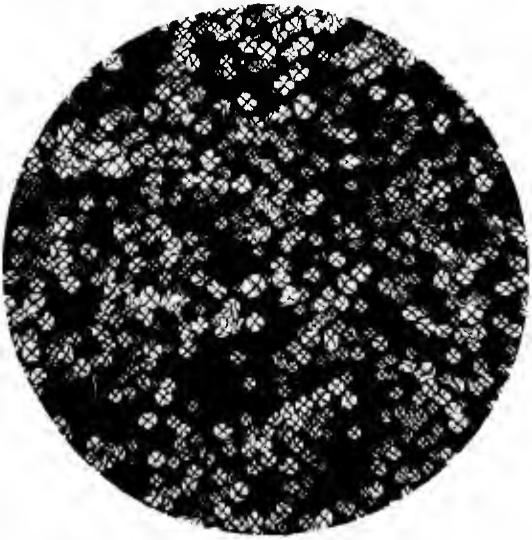
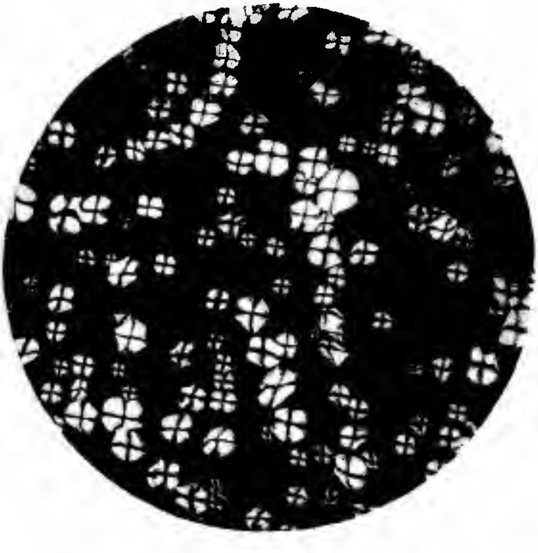
A Hoen & Co., Lithocaustic

Fig 35.



RICE STARCH X 150

Fig. 36.

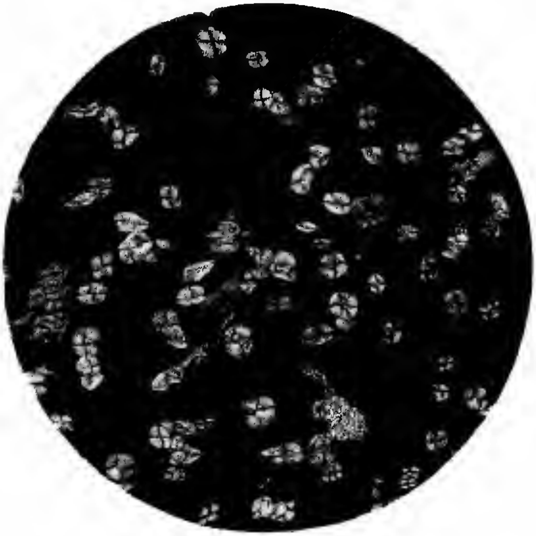


RICE STARCH X 450

PHOTO BY CLIFFORD RICHARDSON.

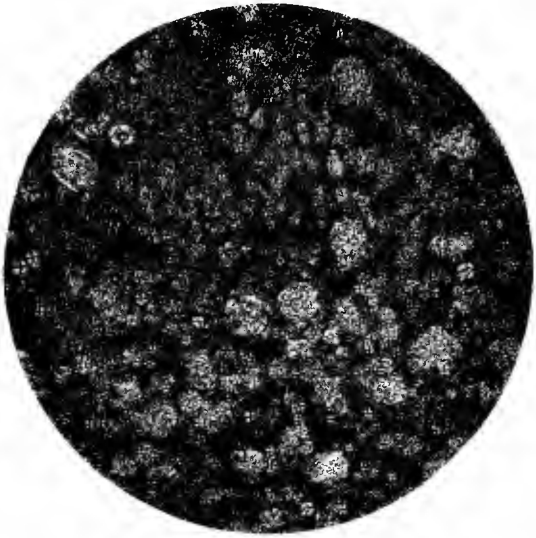
A Hoen & Co., Lithocaustic

Fig. 37.



BARLEY STARCH x 150

Fig. 38.

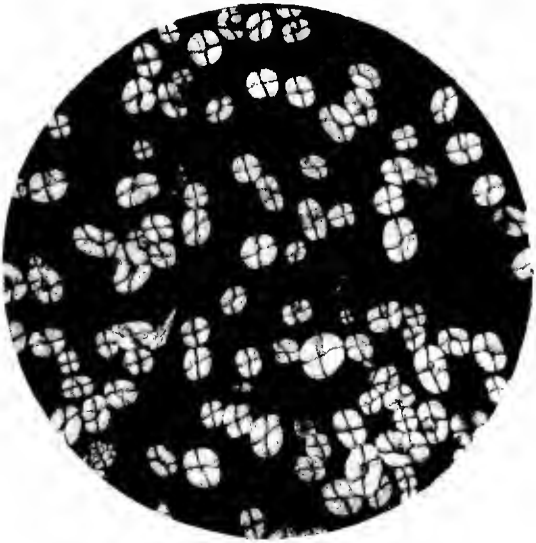


OAT STARCH x 160

PHOTO BY CLIFFORD RICHARDSON.

A Hoen & Co., Lithocaustic

Fig. 39.



BEAN STARCH X 145

Fig. 40.

PEA STARCH X 145

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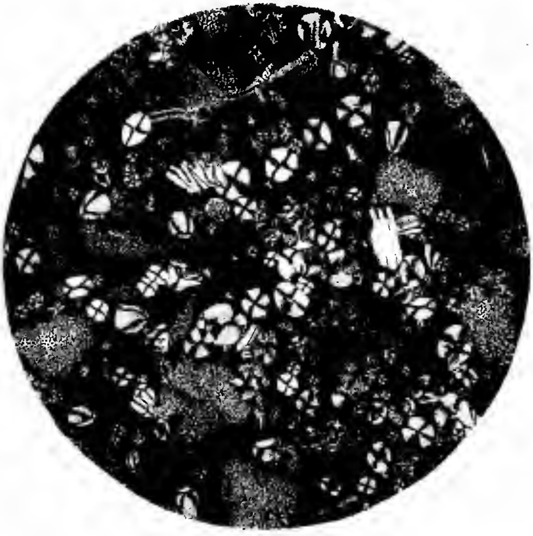
Fig. 41.

GINGER STARCH X 145

Fig. 42.



GINGER ADULTERATED X 145

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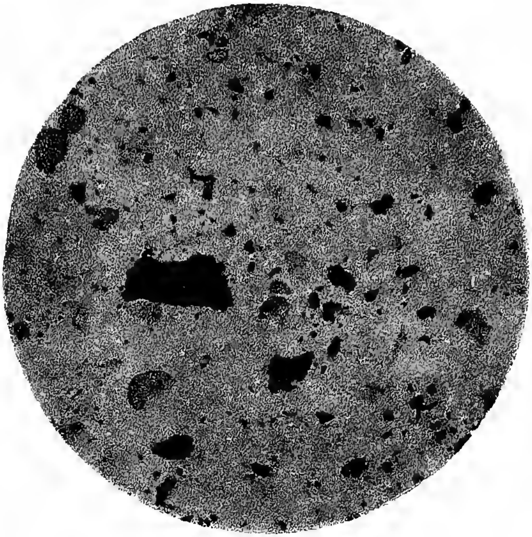
A Hoen & Co., Lithocaustic.

Fig. 43.



BLACK PEPPER P. D. X 45

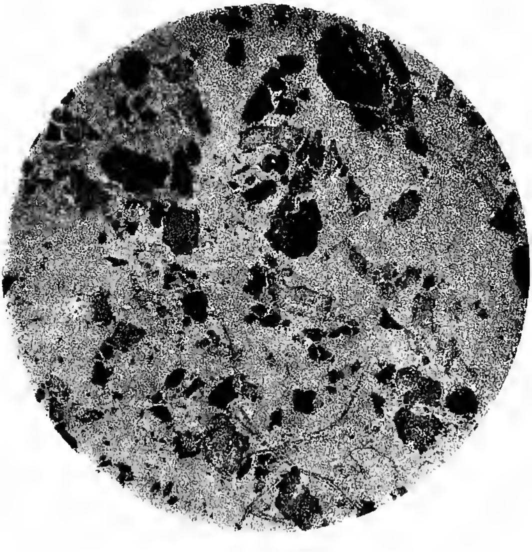
Fig. 44.

PEPPER ADULTERATED X 45

PHOTO BY CLIFFORD RICHARDSON.

A Hoen & Co., Lithocaustic

Fig. 45.



CINNAMON ADULTERATED

PHOTO BY CLIFFORD RICHARDSON.

A Hoen & Co. Lithocaustic

Fig. 46.

CINNAMON x 45

Fig. 47.

CASSIA x 45

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Fig. 48.

CAYENNE

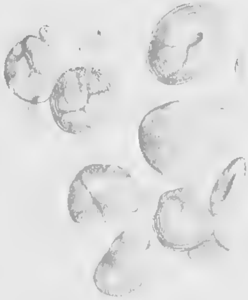
Fig. 49.

DAYENNE ADULTERATED

PHOTO BY CLIFFORD RICHARDSON.

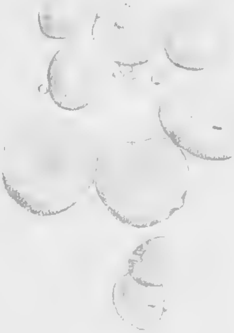
A Hoen & Co., Lithocaustic

FIG. 50



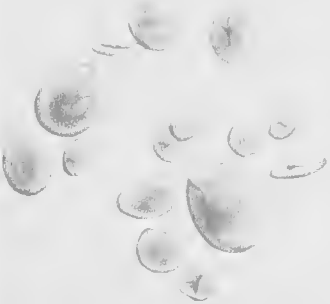
WHEAT

FIG. 51



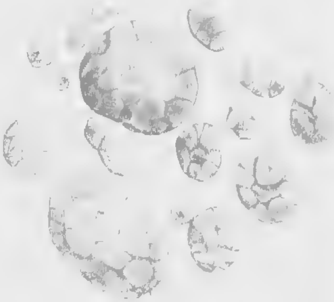
BARLEY

FIG. 52

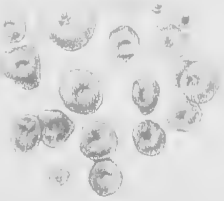


RYE

FIG. 53



OATS



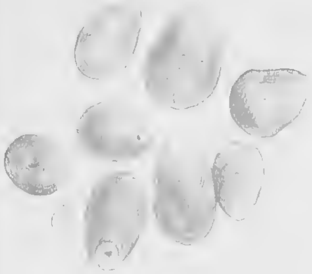
CORN

FIG. 55

RICE

DRAWN BY GEO. MARX. A Hoen & Co., Lithocaustic

FIG. 56



MARUNTA.

FIG. 57

POTATO

FIG. 56

GINGER

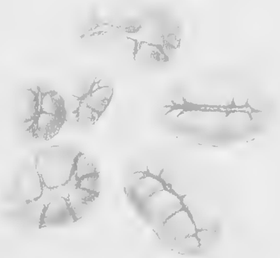
FIG. 59

SAGO

FIG. 60

PEAS

FIG. 61



BEANS

DRAWN BY GEO. MARX.

A Hoen & Co., Lithocaustic

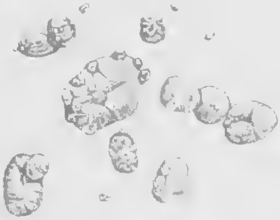
FIG. 62

BUCKWHEAT

FIG. 63

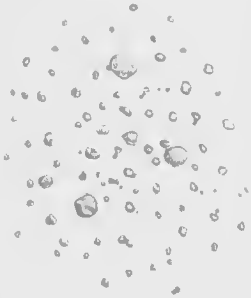
TURMERIC

FIG. 64



NUTMEG

FIG. 65



PEPPER

FIG. 66

CINNAMON

FIG. 67

CAPSICUM

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