



# CHEMICAL AND PHARMACEUTIC MANIPULATIONS.

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# CHEMICAL AND PHARMACEUTIC MANIPULATIONS:

A MANUAL OF THE MECHANICAL AND CHEMICO-MECHANICAL • OPERATIONS OF THE LABORATORY,

CONTAINING

A COMPLETE DESCRIPTION OF THE MOST APPROVED APPARATUS, WITH INSTRUCTIONS AS TO THEIR APPLICATION AND MANAGEMENT BOTH IN

# MANUFACTURING PROCESSES,

AND IN THE MORE EXACT DETAILS OF

ANALYSIS AND ACCURATE RESEARCH. -

FOR THE USE OF CHEMISTS, DRUGGISTS, TEACHERS AND STUDENTS.

BY

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PRACTICAL AND ANALYTIC CHEMIST, AUTHOR OF "APPLIED CHEMISTRY," ETC.

#### ASSISTED BY

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WITH FOUR HUNDRED AND TWENTY THREE ILLUSTRATIONS.

# R

PHILADELPHIA: LINDSAY AND BLAKISTON. 1849.

#### "The House that Jack Bullt."

One of the best travesties on the old nursery tale the foil wing from an exchange:

- he Write House-This is the house that Sam
- 100.000 Th s is the malt that lay in the house
- ames Buchanau—This is the rat that are the malt, that lay in the house that Sam built. . A. Douglas—This is the cat that killed the rat.
- that sie the malt, that lay in the house that
- horn, that tossed the d.g, that worried the cat, et.
- ew York Express-This is the maiden, all for-lorn, that miked the cow with the crumpled horn, that, etc.
- ournal of Commerce-This is the man, all tat-tered and torn, that Ess.d the maiden, all
- forloro, that, etc. Iew York Observer-This is the priest, all shaven and shern, that married the man, all tattered
- and t rn, unto the maiden all foriorn, etc. ndependent---This is the cock that or wed in the morn, to waken the prizet, all shaven and shorn, that married the ma , all to tered and torn, etc.
- be Lincoln-This is the hunter with trampet and hun, that owned the cock that crowed in the more, to waken the priest, all shaven and shorn, that married the man, all tattered and torn, unto the maiden all forlorn, that milled the cow with a crumbled horu, that to seed the dog, that w rried the cat, that killed the rat, that are the mult, that lay in the house that

. B.'s Farewell to the White House. Farewell to the land where the gloom of my glory Arose and o'ershadowed the earth with her

the abandous me now, but the page of her story,

- The foulest and blackest, is fill'd with my shame have war'd with the Yankees, who vanquished
- When the scent of the "darkey " allared to his
- have coped with the free who despise me thus lone'y.
  - The last traitorous " Dough-face " who'll sit in this chair. M.

LINCOLN'S MAJORITY IN NEW YORK .- The Tribune of the 12th foots up Lincoln's majority in that State about 51,777. The rural districts loom up. St. Lawrence Co. gives 7,214 Republican majority !

MARRIED.

On January 3rd, 1861, by Rev. S. Searls, 5. COLLINS, PRINTERS. at the residence of the bride's father, in Dartford, Dr. H. L. BARNES and MISS NELLY E. CODY, all of Dartford, Wis.

# OUR PUBLIC SCHOOLS.

[The following beautiful song, by Mrs. C. H. GILDERSLEEVE, was sung at the late Teachers' Convention at Buffalo.]

A song, a song for public schools, Our people's proudest glory,

- And while we sing, the nation's stars Grow brighter at the story,
- And lighter float those restless folds,
- And higher still we follow; And scorn a name whose only sound, Like ringing gold, is hollow.

Then free as air shall knowledge be, And open window's portals,

To every thirsty, earnest soul, Who longs to be immortal.

Here rich and poor stand side by side To quaff her poorest chalice,

- And never dream that deathless names Belong to cot or palace.
- The light of truth shall guide us on, When glory lies before us,
- And "Right makes Might" emblazoned on The banner waving oe'r us.

A song, a loud, exultant song Shall ring from sea to prairie, And tell the world that MIND not GOLD, Shall make our stations vary.

LADELPHIA :

#### BY OLIVER WENDELL HOLMES.

e count the broken lyres that rest, Where the sweet wailing singers slumber, t o'er their silent sister's breast The wild flowers who will stop to number ? few can touch the magic string, And noisy Fame is proud to win them; is ! for those that never sing; But die with all their music in them !

y, grieve not for the dead alone Vhose song has told their heart's sad storyep for the voiceless, who have known be cross with ut the crown of glory ! where Leucadian breezes sweep "er Sappho's memory-hannted pillow, where the glistening night-dews weep "er nameless sorrow's church yard pillow

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# EFACE.

"er nameless sorrow's church yard pillow, hearts that break and give no sign we whitening lips and fading tresses, Death pours out his cordial wine, [sesow-dropped from Misery's crushing press nging breath or echoing chord every hidden pang were given, the futility of which is principles are supported by facts riment.

t endless melodies were poured; sad as earth, as sweet as heaven ! ence, is the only sure way of pursuing investigation, for it is by conclusions, and not hypotheses, that we can show the composition of bodies, or the principles which govern their reactions.

> To realize, therefore, for chemistry the simple definition of a science,—to render it "a system illustrated and proved by experiment," it is requisite for us to acquire some proficiency in those mechanical operations by means of which chemical changes are produced, observed and estimated.

> This accomplishment in manipulation,—this expertness in *handling* implements, it is true, demands practice and experience; but though the student cannot become an adept in the art solely by the aid of written directions, yet much may be communicated which will lighten labor and facilitate him in the attainment of skill and accuracy.

24170

#### PREFACE.

Such has been the author's object in preparing the present work, which in its arrangement is designed to lead the uninitiated step by step into the mysteries of manipulations; and in which he has endeavored with less regard to elegance of " diction than to perspicuity, to present plainly and clearly such information as is best calculated to give familiarity with the construction, arrangement and uses of apparatus.

Positive originality can scarcely be expected in any account of the well known appliances of the chemical art. The author has, however, while availing himself freely of the knowledge of others, endeavored to combine with it, as much as possible, personal experience, and to present descriptions of new and important forms of apparatus, with practical suggestions of a novel character.

In confessing indebtedness to other chemists, the author acknowledges his obligations to Prof. J. B. Reynolds for the whole of the chapter upon "Analysis by Polarization of Light," a subject which the writer's skill and experience have well qualified him to illustrate.

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UNTERTOR OF A. LALORATORY.

# THE LABORATORY.

# CHAPTER I.

THE Laboratory is emphatically the work-shop of the chemical operative; and chemical manipulation may be termed the practice of the science. A convenient arrangement of the first is no less desirable, for the success of operations, than a proficiency and skill in the latter is indispensable. New facts in science are mainly developed by experiment; and as chemistry is a purely experimental science, in every course of research, as well in the most ordinary experiments as in the more delicate manipulations of analyses, the surest basis of accurate conclusions is an exact and skilful manipulation coupled with correct reasoning. This exemplification, by the hands, of the conceptions of the mind, is, therefore, an art of the highest importance in the pursuit of chemistry.

The laboratory should be appropriately fitted, and arranged with a view to the easy prosecution of chemical investigation in all its several branches; and being the place where most of the operator's time is so profitably and pleasantly employed, no little regard, in its appointments, should also be given to personal comfort and convenience. We do not recommend extravagance in its furniture and paraphernalia, or yet a too rigid economy, for though a stinted apparatus may, by ingenuity and skill, be rendered subservient to the requirements of the science, a liberal endowment is far preferable, and more conducive to rapidity of progress and accuracy of results. In the present advanced state of the mechanic arts, it is doubtful economy to consume time in constructing contrivances, when the most convenient apparatus may be readily procured at the lowest rates. Moreover, a familiarity with the use of good tools originates habits of correct and

delicate manipulation, and will afford, to the experimenter, a proficiency enabling him, in any emergency, to substitute available material for deficient apparatus; whilst in working upon rare substances, the minutest quantity will be no bar to his skill and accuracy in bringing out nice results.

We do not, in the following suggestions, provide for such a laboratory as is suitable for a public institution, because the adaptation of one of that extent would be attended with an expense inconsistent with individual means, as there are many auxiliaries required in class experiments which may readily be dispensed with in an ordinary laboratory; but, we present an apartment economically and conveniently arranged for private research, with space and furniture enough, with some slight multiplication of the apparatus, for two, four, or more experimenters.

In the construction of a laboratory, particular attention should be paid to the lighting and ventilation of the apartments, both in regard to the health and comfort of its occupants. The preferable mode of lighting is by side windows, and for many reasons; it is more advantageous in examining the behavior of re-agents to solution, especially in those instances of delicate testing, where the result is determinable by the formation of flocculæ, faint cloudiness, or by slight transmutation of color. By elongating the windows to nearly the whole height of the apartment, we obtain the magic influence of the solar rays, now known to be so effective in inducing chemical changes unattainable by other means. The skylight arrangement has the double disadvantage of presenting nuclei for the accumulation of dust, and being subject to frequent breaches by accident or storm. Ventilation may be accomplished thoroughly by means of counterpoised windows and stationary hoods.

The laboratory apartment should be sufficiently spacious to afford a separate position for each of the requisite utensils. Too much crowding of apparatus engenders liability of damage, and is, besides, inconvenient, for nowhere than in a laboratory is there more necessity of a strict observance of the rule, "a place for everything, and everything in its place." Hunting up mislaid apparatus consumes time, and the delay thus occasioned, in many instances, may be the means of serious detriment to important operations.

A roomy apartment on the first floor of a building is best



GRADUNID-PLAN OF A LABORATORY.

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ratus, to keep the side door i (7 feet high, and 2.10 wide) leading into the main apartment constantly closed. The front of this wing is, as to windows, identical with that of the office. The free admission of light, which is effected by means of the two long front and three elevated side windows, is as requisite in this as in the operating room. The chimney of this apartment occupies the centre of the outer wall, and receives the main flue, furnishing draft to the main furnace and two lateral branches. Of these two branch flues, both of which have circular openings with movable tin stopples, one is for the reception of the smoke-pipe of the still furnace E, and the other for that of the steam generator F; or, when not in use otherwise, for the portable, blast, and other furnaces. These flues are fitted with dampers to regulate the draught; and the circular opening, when not occupied with apparatus, or as vent holes for the dispersion of noxious vapors, should be kept covered so as to preserve unimpaired the draught of the main furnace. In the arrangement of a room, for laboratory purposes, wherein the flues are not convenient, they must be substituted by stove-pipes.

The Furnace.—The furnace G, which is in constant use for the ordinary operations of the laboratory, occupies the centre of the outer wall. That of most convenient construction is described by Faraday, to whom we are indebted for both our description and figures.

"Being in constant requisition as a table, it should be about 34 or 35 inches in height. The brick work should measure 36 by 20 inches, and the iron plate, including sand-baths, 40 by 28 inches. A warm air chamber may be built in the walls beneath the flue. Projecting spikes should be fastened into one or two sides of this chamber, to hold a temporary shelf when required.

"Precipitates, filters, and other moist substances put into such a chamber, are readily and safely dried. The hot air causes evaporation of the water, whilst the current removes the rising vapor. The chamber is very useful in effecting the slow evaporation of liquids, and also for hot filtrations, when the entering current of air is of a temperature sufficient for the purpose.

"The principal part of this furnace is necessarily of brickwork, only the top plate with the baths and the front, being of iron. The front is a curved iron plate, having two apertures closed by iron doors, one belonging to the fire-place, and the other to the ash-pit. It is 34 inches high, and 14 inches wide. The ash-hole door moves over the flooring beneath;



the bottom of the fire-place door is 22 inches from the ground, and the door itself is  $8\frac{1}{2}$  inches by 7. This front is guarded within at the part which encloses the fire by a strong cast-iron plate, having an opening through it corresponding to the door of the fire-place. It has clamps attached to it, which, when the furnace is built up, are enclosed in the brick-work.

In the setting or building of the furnace, two lateral brick walls are raised on each side the front plate, and a back wall at such a distance from it as to leave space for the ash-hole and fire-place; these walls are lined with Welch lumps, where they form the fire-chamber; two iron bars are inserted in the course of the work to support the loose grate bars in the usual manner, the grate being raised 19 inches from the ground. The side walls are continued until of the height of the front. and are carried backward from the front in two parallel lines, so as to afford support for the iron plate which is to cover the whole. The back wall of the fire-place is not raised so high as the side walls by six inches and a half, the interval which is left between it and the bottom of the sand-bath, being the commencement of the flue or throat of the furnace. In this way the fire-place, which is fourteen inches from back to front, and nine inches wide, is formed, and also the two sides of the portion of horizontal flue which belongs to the furnace, and is intended to heat the larger sand-bath. The bottom of this part of the flue may be made of brick-work, resting upon bearers laid on the two side walls, or it may be a plate of cast-iron resting upon a ledge of the brick-work on each side, and on the top of the wall, which forms the back of the fire-place. When such an arrangement is adopted, the plate must not be built into the brick-word, but suffered to lie on the ledges, which are to be made flat and true for the purpose; for, if attached to the walls, it will, by alternate expansion and contraction, disturb and throw them down. The ends of the side

#### THE FURNACE.

walls, forming as it were the back of the furnace, may be finished either by being carried to the wall against which the furnace is built, or enclosed by a piece of connecting brickwork, to make the whole square and complete, or a warm air cupboard may be built in the cavity beneath the flue, and the door made to occupy the opening between the walls. Occasionally the flue may be required to descend there, and pass some distance under ground. These points should be arranged and prepared before the plate constituting the top of the furnace is put on to the brick-work, so that when the plate with its sand-baths are in their places, they may complete the portion of horizontal flue by forming its upper side.

The size of this plate is the first thing to be considered, and having been determined upon, from a consideration of the situation to be occupied by the furnace, and the places of the sand-baths also having been arranged; the brick-work must then be carried up, so as to correspond with these determinations, and with the plate itself, which in the mean time is to be cast. The sand-baths and the plate are to be formed in The bath over the fire is best of a circular separate pieces. form, and of such diameter that, when lifted out of its place, it may leave an aperture in the plate equal in width to the upper part of the fire-place beneath; so that a still, or cast-iron pot, or a set of rings may be put into its place over the fire. The other sand-bath must be of such a form as to correspond with the shape and size of the flue beneath. These vessels are to be of cast-iron, about three-tenths of an inch.

thick; their depth is to be two inches and a half or three inches, and they are to be cast with flanches, so as to rest in the corresponding depressions of the plate that the level of the junctions may be uniform. This will be understood from the accompanying section of the furnace, given through the



line AB of the view. It is essential that these sand-baths be of such dimensions as to fit very loosely into the apertures in the plate, when cold, a space of the eighth of an inch or more being left all round them, as shown in the section, otherwise, when heated, they will expand so much as entirely to fill the apertures, and even break the plate. The plate itself should be half an inch thick.

When the plate and its sand-baths are prepared, and the brick-work is ready, the furnace is finished by laying the plate on the brick-work, with a bed of mortar intervening. If the walls are thin, or any peculiarity in their arrangement occasions weakness, they should be bound together, within by cranks built into the work, and without by iron bands. The alternate changes of temperature from high to low, and low to high, to which the furnace is constantly subject, renders it liable to mechanical injury, in a degree much surpassing that which would occur to a similar piece of brick-work, always retained nearly at one temperature." The square space enclosed by the fire-place and flues may be converted into an excellent drying or warm air chamber if desired.

Cast-iron is the best material for these baths, for, though liable to be cracked when first heated, by their unequal expansion in different parts, they do not warp and assume the irregular and inconvenient shapes that wrought iron acquires under similar circumstances.

"These baths should have washed sea-sand put into them; it is heavy, and occasions no dust when moved, whilst, on the contrary, unwashed and bad sand contains much dirt, and occasions great injury in experimenting. A piece of straightened iron hoop, about twelve inches in length, should lie on the furnace, as an accompaniment to the baths, being a sort of coarse spatula with which to move away the sand.

The circular sand-bath is frequently replaced by a set of



concentric iron rings, or a cast-iron pot. The rings are convenient for leaving an aperture over the fire of larger or smaller dimension, according as a smaller or larger number are used at once; and being bevelled at the edges, fit accurately into each other, without any risk of becoming fixed by expansion. The external one, like the sand-baths, should be made smaller than the depression in the furnace plate in which it rests. The iron pots are of various sizes, and are adapted to the furnace by means of the rings; a red heat is easily obtained in them for sublimation."

In many instances, where economy is of prime importance, the foregoing sand-bath can in a measure be substituted by an ordinary cylinder stove, the pipe of which leading into a four sided sheet iron box, divided into flues by partitions, imparts its heat which eventually passes into the chimney. The top of this box when covered with sand, forms the sand-bath. That portion of its surface immediately over the first flue, is the hottest. The remote or cooler end, is best adapted for gradual digestions, evaporations, &c.; and so by these flues there is a means of graduating the temperature of the bath. The top of the stove itself being directly over the fire, makes an excellent bath for those operations requiring a higher temperature.

This arrangement, or the still more economical gas bath (Fig. 27) described at p. 48, renders necessary the use of Luhmè's or Kent's portable furnace for fusions, crucible or other operations requiring a very high heat, but this involves no additional expense, for such an implement is indispensable for other laboratory purposes.

The steam generator (Fig. 10) when used as a stove for heating the apartment, answers equally well to heat the bath, it being only necessary to conduct its smoke-pipe into the iron box instead of leading it directly into the chimney.

To prevent contamination of the atmosphere of the apartment, by admixture with the deleterious fumes evolved during the various operations of digestion, fusing, melting, heating, and evaporating in progress upon the sand-bath and in the furnaces, there should be firmly fastened to the ceiling and immediately over its surface, extending beyond its superficies some four inches all around, a sheet-iron hood, of form at the base corresponding with that of the top of the furnace. The barrel of this hood may pass either directly through the ceiling and roof into the atmosphere,\* or else be

\* When the external atmosphere is colder than that within, an air-vent overhead does not thoroughly relieve the room of its noxious vapors, for the cold air rushing in depresses them,—even within the sphere of respiration, and thus prevents their ascent and consequent escape through the hoods. Dr. Murray's very simple and effectual plan of ventilation, is to conduct a funnel-mouthed pipe through the ceiling into a chimney where a constant fire is maintained. To provide against the entrance of smoke by reason of imperfection of draught, formed into an elbow, leading into the main flue of the chimney. In either case the draft must be thorough, so as to afford a free egress of the fumes into the atmosphere without. It



should also be immovably fixed by rod iron stretchers, and well payed over with plumbago paint, which is a resistant of the corrosive effect of the laboratory fumes, and thus prevents the destruction of the metal. The fixture is represented by Fig. 9. It should descend as near to the surface of the bath as convenience of manipulation will allow; and to prevent any accumulation of dirt in the interior, it should be frequently brushed out with a

soft brush; and for protection to the vessels on the sand-bath, against falling particles, the top of the furnace should, during the operation, be covered with paper. It is advisable at all times, independently of the foregoing suggestion, to keep each vessel covered with plates or clean white paper, which, while protecting against dirt, offers no impediment to the processes of evaporation, digestion, &c. If the hood, instead of being fixed is counterpoised, so as to admit of ready depression or elevation at will, it is a little more convenient; but that arrangement has the disadvantage of liability to accident, for a carelessness in fastening the suspension cords may create a very annoying damage. Of course, this mode of hanging the hood can only be adopted where the barrel or pipe is straight, and leads directly through the roof; then to protect the exit hole from the wear and tear consequent upon the abrasion of its circumference, it should be fitted with an earthen ware cylinder'; and furthermore, to prevent the entrance of rain through the

the pipe should be carried to the top of the chimney. The uniformly high temperature of the chimney keeps the pipe constantly hot, and thus the mephtic vapors within the room will be disengaged. By arranging the barrel of the hood as thus directed, an equally effectual disengagement of vapors may be obtained. slight openings, there should be a spreading flange around the protruding portions of the barrel of the hood, near the roof.

The Steam Generator.—To the right of the furnace, at a convenient distance, is the portable steam generator F, with its smoke pipe leading into the circular opening of the lateral flue opposite. This is a patent invention by C. W. Bently, of Baltimore, Md. It has a stove-like form, is compact, requires no brick work and but very little fuel, and can be set up and removed at will, when it is desired to occupy

the flue with other apparatus. The only fixtures requisite, in addition to the machine, are feed pipes to convey the water, and conduits for the passage of the steam. It is a most convenient apparatus for the laboratory, being alike handy for economically supplying hot water to all parts of the building, and for boiling substances, where the direct admission of steam is preferable; and also for heating the steam baths in the range a little to its left. This mode of applying heat, having the great advantages of safety, convenience and regularity, is absolutely requisite in many cases where the naked fire does not offer that uniformity of temperature necessary to the inalterability of certain substances under process. Fig. 10 represents By means of couthe apparatus. pling screws and flexible lead pipe, (Tatham's most preferable, being smooth within,) the steam may be

Fig. 10.

carried to any reasonable distance in any direction, thus affording great facility in many operations; as the loss by condensation in thus conveying it is inconsiderable. In very cold apartments, however, when the conduit pipe is of any great length, it may very properly be enveloped with woolen listing or other bad conducting materials. Unless this machine is kept in constant use as a heater for the building and

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the sand bath, as suggested at page 31, wood is *the more* preferable fuel, as it admits of ready ignition, and enables a speedier generation of the steam than could be obtained with coal. The lower cock in the figure connects with the feed pipe. The three smaller cocks above, and placed equi-distant from each other, are try cocks, to ascertain the height of the water, by which its supply must be accordingly regulated. The steam conduits are coupled by a cock fitted to the top of the generator.

The door in the lower part is for the introduction of fuel into the fire hole.

Steam-baths.—Immediately to the right of the generator, and affixed against the back wall of the room, as at ll, plate 2, are the steam-baths, mounted in a wooden frame work (Fig. 11). Two or three are as many as necessity calls for in the laboratory. They are of copper, and double bottomed; the inner jacket of one may be of smooth copper, and the other of tinned copper or sheet lead. The larger of the two may have a diameter of thirteen inches and a depth of twenty inches, the smaller being each way five inches less in size. Fig. 11 represents the apparatus. There can be a third,



with very little additional expense of money or room, and this should have its inner jacket of thin cast iron with porcelain lining, as being more suitable for those operations which are corrosive of metallic vessels. These latter are made to order by Savery, the former by Hammet and Hiles, of Philadelphia. The outer jackets b b b are invariably of copper.

Immediately over the frame work, which is stationary, resting and fastened against the back wall, is a welded wrought iron steam conduit B, forming the main feeder for the baths a a a. The supply of steam, which is conveyed to each through a pipe d, connected with the main feeder and fastened to the back of the outer jacket b, is regulated by the cocks f and c. The safety valves are supported by uprights  $e \ e \ e$ , firmly fixed to the floor beneath. The stop cocks  $c \ c \ c$  render each kettle independent of the others, so that the use of one does not necessarily compel all to be in operation.

This apparatus is very convenient for exhausting vegetable matters, such as dye woods, plants, &c., of their matter soluble in water, and whose active principles are liable to be damaged by fire. The saving in fuel and time, the perfect freedom from waste steam, the power of regulating the heat, are only a few of the advantages of this mode of boiling over the old plan of heating in open kettles over the naked fire.

Moreover, when the exhaustion is complete, the heat may be discontinued by merely stopping off the steam with the cock c. By another connection with the hydrant, enabling a current of fresh water as soon as the steam is turned off, the apparatus is converted into a refrigerant, and its contents may be cooled as suddenly as desired.

Near to the steam series, are two steaming cisterns of an half-barrel capacity each. One may be of deal or oak wood and iron bound, the other of blue stone-ware, from the Baltimore pottery. These tanks are mounted upon pedestals, and being readily handled for filling, emptying, and cleansing, are very convenient for those operations where the direct application of steam is necessary. A flexible leaden pipe from the main conduit, leads the steam directly into the vessels, and produces a uniform ebullition. The form of these tanks is similar to that of butter or meat tubs. To prevent the diffusion of the steam through the apartment, the vessels must be kept covered during the operations of boiling.

The Still.—On the left of the furnace, as at E H, Pl. 2, and occupying the same relative position there as the generator on its right, are the still and refrigerant, which are indispensable utensils, both for a supply of pure water for analyses, &c., and for the many distillatory operations connected with chemical research. In its construction there should be particular regard to compactness, so that the implement may combine the double advantage of a naked and water bath still. For convenience and economy of room, we prefer that this apparatus be movable, and therefore recommend, as a substitute for a brick wall bed or setting, a portable stove-like cylinder of thick sheet iron. The fire door is as shown in the figures below, and in order to prevent the overheating of the iron cylinder, the part which contains the fire should be lined with a refractory earthen cylinder, of about two inches thickness, as at b and c, in Fig. 12. The smoke pipe leads into the circular opening of the op-



posite flue. The body of the still rests upon the rim of this furnace at a, by its flange, which surrounds it immediately below its handles. It is shown by C, Fig. 13. Its dimensions should be so much less than those of the furnace, as to leave sufficient heating space around its sides and bottom. The material of the still is copper, and the

joints are rounded so as to give every facility in cleansing. Moreover, round edges are less liable to become bruised than



Fig. 13.



the angular. For the distillation of substances indestructible at high temperatures, this still is applicable over the naked fire, but for more alterable bodies, the intervention of water is necessary, and so, accordingly, an inner tinned copper or enameled iron jacket is provided. The form and position of this jacket are shown at  $\hat{B}$ , by the dotted lines in Fig. 13. It is a straight cylinder with convex bottom, and a broad rim, serving also as a flange or rim for its support in the still. Its dimensions are four inches in diameter and eight inches in depth less than those of the still. The head or capital, which should be of tinned copper, or, preferably, of pewter, is shown at A. The rim is made to fit the mouth of either the still or water bath, and hence the same head answers in both naked and bath distillations. The beak conveys the vapors accumulating in the capital, into the refrigerant or condenser, which consists of a pewter worm Fig. 14, encased in a wooden tub kept constantly supplied with cool water through the pipe e. The water pipe which carries off the heated water displaced by the cold water, runs from the top of the tub, and has its exit into the sink, or through the wall, into the gutter. These two pipes are better of lead. The vapors in passing through the worm are condensed and drop as a liquid into a receiver, which is placed beneath the outlet pipe near the bottom of the tub.

To convert the apparatus into a water bath, (for in many distillations the temperature must not exceed the boiling point of water or a saline solution,) it is only necessary to charge the outer jacket, or still, with the proper quantity of liquid, and then to insert the inner casing B, which slides into the mouth and fits tightly.

In the distillation of flowers, roots, and other substances, in the naked still, a too close contact with its heated sides and bottom renders them liable to injury by scorching, and

therefore it is necessary to have a strong wire stand with one or two cullendered shelves upon which to place the material. The lower shelf h being an inch or two from the bottom of still, prevents all liability of contact between it and the material. This apparatus is shown by Fig. 15. When the still is not in use for its legitimate purpose, by removal of the wire

Fig. 15.



shelving, it becomes an excellent kettle for any of the ordinary boiling operations.

In the blank space of the wall to the left of the front entrance, stands a deal wood cask, with wooden spigot, mounted upon a stand of convenient height. This serves as a reservoir for distilled water, and the opening for pouring in the water must be kept tightly closed to prevent the admission of dust or absorption of gases.

The Sink.—In the corner of the room to the right of the refrigerant, is the sink. Its position will be better understood by reference to I, Pl. 2. As it is necessary that the laboratory should be abundantly and constantly supplied with water for cleansing, distilling, and many other operations, it is better in those cities where the water is supplied by public water-works, to make an attachment to the main conduit, and lead the water through a lead pipe directly into the laboratory, and immediately over the sink. The only arrangement necessary is a stop-cock at the termination of the pipe, to regulate the flow of water. Fig. 16 represents a sink thus arranged. The trough should be of wood and lined with



sheet lead, which metal is preferable to zinc; because less liable to corrosion by acids, for the formation of holes, by amalgamation with mercury, can be avoided with a little care in washing vessels containing residua of it, or the solutions of its salts. The floor beneath, to a certain extent around the sink, should also be covered with sheet lead, otherwise its continual dampness from the splashing water would endanger the health of the operator. When the introduction of water by conduit is impossible, it is necessary to erect immediately over the sink a strong iron-bound oaken reservoir with cover, which must be daily filled with buckets from the neighboring pump. In either

case, the exit-cock should be fitted with one of Jennison's filters, a small metallic casing, Fig. 17, containing a stratum of crushed quartz, which arrests the suspended impurities of the water during its percolation through. If it is not convenient

## THE LABORATORY-THE STILL.

to provide one of the above filters, an economical, but slower and less convenient one can be made of a common red earthenware flowerpot by covering its bottom interiorly with a linen cloth and filling it with coarse white sand. The waste-pipe, which must be constructed so

as to admit of the *free* egress of the waste-water, passes into a drain which conveys its charge into cess-pools or tanks lined with brick, and sunk into the ground. As the emanations of foul air from these pools are noxious, they should be placed some distance from the building, and kept well covered. If the situation be favorable, the drains should empty themselves into a gutter or some running stream, which, in conducting away the foul matter, would relieve the air of the apartments of its noxious effluvia.

In order to prevent the entrance of any unpleasant smell through the apertures by which the water goes down, there should be a *bell stench-trap* at the commencement of the drain. This addition, which will be furnished to order by the plumber who constructs the sink, has the additional advantage of retaining particles

of solid matters that may fall down. It is shown by figures 18, 19, for which we are indebted to *Webster's Encyclopædia*. "Fig. 18,  $a \ b \ c$  represents the section of a portion of a hollow cone of metal, having a short pipe in the middle,  $b \ d$ ; and water is put into this cone up to the level  $a \ c$ . A loose perforated cover e is made to rest on a shoulder on the top of the cone, and this cover is perforated with two circles of holes; on the lower side of this cover a hemispherical cup is fixed, the edges of which dip under the surface of the water.

When water of any kind is thrown on the cover, it passes down through the holes, and finds its way under the edges of the inverted cup, down Fig. 19.

through the tube d, and so into the drain; but if any foul air should come back the same way, before it gets out it would have to pass through the water; but from its levity it lodges in the top of the hemi-





spherical cup, and cannot descend through the water, unless more pressure is exerted than is usually the case; hence the cup dipping into the water is a complete trap or stop for the air, and effectually hinders any bad smell or noxious effluvia from coming up from drains, which, indeed, should never be without this simple but useful contrivance. These traps likewise prevent the intrusion of rats, &c. This apparatus, however, is sometimes liable to be deranged by neglect or bad usage; and it is proper to construct another kind, of brickwork. Somewhere in the course of the drain let there be sunk a small square well, Fig. 19, g g, built round with bricks laid in cement, and plastered on the inside with the same, so as to be completely water-tight and to remain always filled with water. Across this well let there be a piece of paving stone so fixed that its top may touch the cover of the drain, and its lower edge dip below the surface of the water in this trap or well. On the same principle as the bell trap, no air can pass along the drain, it being stopped by the water below the stone."

As all the cleansing operations are performed at the sink, it is necessary that it should be fitted with several shelves, in addition to those which may be arranged by its sides. To afford free egress to the draining water, those which are to hold the glass-ware had better be cullendered, and upon one, for the safety of the test tubes and other hollow apparatus of too



small circumference to stand upright alone, there should be a series of draining-pins as shown by Fig. 20. A rack of horizontal pegs, for draining retorts and other irregular-shaped apparatus, might also be conveniently arranged upon a part of the space. For draining vials and small flasks, an upright stand fitted with pegs, as shown by Fig. 21, is perhaps preferable to the horizontal rack. A jar of soft and a piece of castile soap should have appropriate positions in the vicinity of the sink; and near by also, say on the back of the door, for the sake of economizing room, there must be two long towels hung on rollers as at Fig. 22. One of these towels is exclu-

sively for the hands, the other for drying the cleansed glass-ware, &c. The other accompaniments to the sink are a coarse towel, a small paint-brush, a bottle of shot, a series of wires, some tow and raw cotton, and a wire instrument for the removal of corks from the interior of bottles. This latter is nothing more



than three plies of stiff wire united together at their upper ends, and bent in angular forms at their lower ends. The paint-brush is for washing out wide-mouthed apparatus, and can be well substituted by a twine-brush of similar shape, and much used in housewifery for washing tea-china. These and the cork wires are to be had at any house-furnishing establishment.

Of the series of wires, one should be stiff and skewer-like, with pointed end, to remove those particles of dirt, tenaciously adhering to bottles, which have resisted the cleansing action of agitation with shot. The remaining wires may be of stiff iron and roughened, or jagged at the ends, in order the more securely to prevent the slipping of the tow or cotton, which is wrapped and tied thereon to facilitate the cleansing of the glasses. The tow or cotton is to be renewed as frequently as is necessary to cleanliness. A portion of the wires may be from  $\frac{1}{2}$  to  $\frac{1}{4}$  of an inch thick, and 16 to 18 inches long. The rest for smaller apparatus may be of proportionally less dimensions. Several long wedge-shaped oaken sticks are also convenient for more effectually applying the cloth or towel, with which they are temporarily wrapped, to the angular spaces at the bottom of the glasses. All of these pieces of apparatus should have appropriate places near to the sink. A series of pegs or nails are very convenient hangers, and two or four cuddies make serviceable receptacles for the tow, cotton, and rags.

In those situations where it is not convenient to introduce the water through a pipe, there must be erected immediately over the sink a strongly braced shelf, as a support to a closely covered deal wood cistern for the reception of water. The water is supplied either by buckets full from a neighboring pump or else is pumped in. In the former case, the position of the sink in the corner, and near to the door, allows great facility in filling it.

Next to the sink, occupying the inner wall spaces on either side of the door, as at m m, Pl. 2, are strong shelving cuddies, racks, and pegs, as receptacles for crucibles, furnaces, iron pots, pans, lead coils, and other apparatus needful in the processes and operations performed in the room.

The corner shelves K, Pl. 2, strongly built, are for the reception of the larger pieces of apparatus. There should also be reserved a wall space for the still, generator, &c., when out of use.

The anvil occupying the position L, plate 2, and resting upon a foot-block, is a most useful implement, and a necessary accompaniment to the tool-chest, upon the opposite side of the room, at n, Pl. 2. This tool-chest, which is shown by Fig. 23, combines in its construction the conveniences of a work-



bench. The vice is affixed towards the end, so as to give full working room. The drawers are receptacles for the requisite tools, among which should be a hammer, hatchet, saw, a chisel of each kind, gimblets, awls, files of the various shapes,

pincers, a soldering iron, a screw-driver, with an assortment of screws, nails, &c. A glue-pot will also be found a necessary addendum. The bench should be about four feet in length, and of height suitable to the comfort and convenience of the operator.

The pedestal o, Pl. 2, occupying the space between the door and left front window, supports a barrel-shaped reservoir of deal wood, or preferably of blue stone, (which can now be had at Maulden Perine's pottery, Baltimore,) for the reception of distilled water, a supply of which should be constantly kept on hand.

A tin match-box, an essential requisite of the furnace room, should have a dry position in some convenient place upon the wall. The charcoal, coke, and sand can either be kept in the cellar, or else in bins occupying the base of the shelving, and resting immediately upon the floor.

A solid oaken pedestal for the iron mortar, and several wooden buckets for general convenience, are also necessary pieces of furniture.

All operations emitting corrosive or disagreeable vapors should be confined, as far as possible, to this room. In passing sulphuretted hydrogen, chlorine, or sulphurous acid through liquids, the vessels should rest either upon a shelf projecting out of the window, or else under a hood which can carry the emanations into the flues, and thus prevent much corrosion of apparatus and discomfort to the operator.

# CHAPTER IV.

#### THE OPERATING ROOM.

BETWEEN the office and the furnace room, and occupying the whole residual floor space C, Pl. 2, of the apartment, is the main operating room (Pl. 1), of dimensions on the plan, 24 by 18.6 feet. In this room are performed all the more delicate manipulations of analysis and experimental research, and hence the necessity of great cleanliness. The arrangement prescribed frees it

entirely from the dust of the coarser operations of the furnace-room, (the door of which should be kept constantly closed,) while the counterpoised windows, of adequate dimensions to afford abundant light, are also capable of maintaining thorough ventilation. In this room are stored nearly all the finer apparatus and materials. The main feature of the apartment is the operating table, which is shown by Fig. 24. Its position (M,



Pl. 2) is against the front wall space between the middle and left window. It may be constructed of pine wood, though cherry or walnut is preferable. At all events, the top, which must project over all around 2 inches, should be either of. these woods or ash, and at least of an inch thickness; glued at the grooves, and grooved and clamped at the cross-grained end, so as to prevent warping or shrinking, either of which creates a great inconvenience to the operator. It is indispensable that the stuff be well seasoned and joined, because any shrinking will leave loop holes for leakings to penetrate into the drawers beneath, and injure their contents. When the top is made and jointed as thus directed, it obviates the necessity of covering with sheet lead, which, though more durable, endangers the safety of glass and other fragile vessels placed upon it. The height of the table proper is 3 feet. Depth 2 feet 10 inches. The length of the top is 4 feet 10 inches. The shelfstand or test case, which slides in grooves, and is fastened to the top of the table by screws, is 30 inches in length, and 30 to 32 in height. The distance between the shelving is unequal, in order to accommodate the different sized bottles. The space between the lower and first shelf may be 10 inches, diminishing gradually upward, so that the interstice between the top and topmost shelf shall not be greater than 5 inches. The shelves may be of light stuff, say 3 inch thickness. The upper drawers should have a depth of  $2\frac{1}{4}$  inches; the lower  $3\frac{1}{4}$  inches. The closets below should be fitted, the one on the right, with movable shelves, the other on the left with rows of wooden pegs, obliquely hung.

This table thus constructed is the operating table of the experimenter, and must be furnished with such apparatus and materials as are in constant requisition, and hence the convenience of the shelving, drawers and pegs, as their receptacles. As it is desirable that the table should not be encumbered with apparatus in unnecessary amount, only those pieces which are of constant use, and required to be at hand, should find an abode within the limits of this table. The general supplies of the laboratory are stored elsewhere, as will be directed hereafter.

One of the upper drawers should be reserved for filters of the different kinds of paper used for the purpose. These may be purchased, already cut, and of the different sizes, neatly put up in boxes, of Kent of New York. If they are made in the laboratory, it is necessary to have a series of circular tins, corresponding with the size of the funnels most in use, by which to shape them.

Another drawer may be reserved for small tubes, rods, pipettes, and glass or porcelain connections. Another for platinum crucibles, spatulas and fine metallic vessels.

The small retorts, bulbs and the like should also have an appropriate drawer. The larger retorts and glass apparatus find appropriate places in the cupboards.

The top drawer to the extreme right should be fitted up in desk-form, and furnished with pen, ink and paper, for the convenience of making rough notes during operations, which are afterwards to be neatly transcribed in a note-book, or "Record of Laboratory Operations," kept especially for the purpose in an appropriate place in the office desk. The valuable information which can in this way be stored up, in a short time amounts to a vast fund, which may, to the great convenience and advantage of the writer, serve as a remembrancer of facts acquired and of errors avoided. A coarse towel should always be an accompaniment to this table, and have a hanging position at its side.

The two lower drawers beneath the closets may be reserved for the more weighty implements.

A leaden funnel, supported by a wooden casing, with its barrel united to a leaden pipe leading through the floor into the street gutter, and placed immediately to the right of the table, would be very convenient for receiving and conveying off the slops from the test tubes. When this arrangement is not practicable, a bucket must be substituted, and emptied daily, for the practice of emptying test tubes upon the floor is



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slovenly and reprehensible, and by keeping it constantly damp, the comfort of the operator is greatly impaired.

A rack with test tubes, Fig. 25, may be considered one of the fixtures of the operating table.

The spirit lamp which furnishes the heat for table operations, and is shown by Fig. 26, will be spoken of more



fully hereafter. When coal gas can be commanded, it is far more convenient and economical, and by a particular arrangement, may be made to yield heat enough for evaporation and ebullition in capsules, and the different operations of digesting in bell glasses, &c. By the use of a large argand burner fixed over the jet of the table blow-pipe, Fig. 30 we can obtain the power of a blast. The admixture of the gas, in this way, with atmospheric air, increases the heat to such an extent as to allow the ignition of precipitates in crucibles, and the almost entire dispensation of FURNACE fires in table operations. The arrangement by which these results are accomplished, so as to avoid entirely the deposition of

carbon on the bottoms of the vessels, is shown by Fig. 27. B



is a cylinder of sheet copper, stretched over the top of which, and fastened by an iron hoop, is a fine wire gauze, covered with fine gravel to protect it from wear and tear. In order to promote a more thorough admixture of the gas and atmospheric air, (which is effected in the chimney,) there is a coarse wire gauze diaphragm c. The gas pipe of flexible

lead depending from, and connected by a gallows screw
h, with the permanent hanger o, terminates in an argand burner d. To prevent a scorching of the table, the burner and cylinder both rest upon a fluted plaster tile. The air enters through the openings in the lower circumference, being drawn up by the upward current of gas, which is let on and regulated by the stop-cock r; and the mixture thus formed passing through the upper fine wire gauze, above which it is ignited, should burn with a bluish flame.

"Where the quantity of gas is too great for the amount of air admitted, the flame will be white and smoky, but by regulating the supply of gas, the due proportion for a blue flame may be easily attained. Now, to obtain a blue flame from a cylinder of large diameter, a considerable quantity of gas will be requisite, and hence an economical advantage is gained by employing cylinders of different diameters. In the same cylinder, also, where different quantities of heat are desired, the lower series of holes may be made large, and a ring of sheet-iron slid over them, by which the quantity of air admitted may be regulated according to the quantity of gas consumed. The cylinders may be  $2\frac{1}{2}$  to 5 inches diameter by 6-8 inches in height; but by introducing several pieces of coarse gauze, c, at short distances apart, the height may be diminished. The highest amount of heat produced by this apparatus is a cherry-red by daylight. For burning off filters in a platinum crucible, a cylinder of  $2\frac{1}{2}$  inches diameter is amply sufficient; but for heating larger vessels, such as capsules, those of 4-5 inches diameter are desirable. This mode of burning the gas presents the advantages of producing any degree of heat as high as a red, of not blackening vessels immersed in the flame, and of avoiding, with more certainty, the fracture of porcelain or glass vessels, from the diffusive character of the flame."

The ring n, sliding upon the rod of the upright stand A, serves as a support for a retort, capsule or crucible. A second chimney g placed over the crucible creates a uniform and constant draught.

The whole of this apparatus is movable, and when the space which it occupies upon the table is required for other purposes, it is only necessary to disconnect it from the hanger, and place the whole aside, to be as readily replaced again when wanted.

The introduction of gas into the room also allows the substi-

tution of an economical table sand-bath (Fig. 28), for the more cumbersome one described at pp. 30, 31. It consists of a copper

Fig. 28.



box B eighteen inches long, twelve inches wide and six inches deep. The top, which is ledged, projects over about an inch and forms the bed for the sand. The door c having a small semicircular opening at its base, is for the entrance of the gas pipe with an argand burner attached, as well also for the supply of air necessary to sustain combustion. The fire thus applied heats the sand on the top. The heated air has an exit through the circular aperture a, after having traversed the interior, which is divided lengthwise by the partition as represented by the dotted lines. The communication between the apartments is by an opening d in the diaphragm. In this way we obtain a graduation of the temperature of the bath. The Swedish chemists improve upon this construction, by annexing an apartment for drying filters and precipitates as well as for keeping liquids hot while filtering.

These, with the test bottles and contents, complete the paraphernalia of the operating table, and so we proceed to describe the next most important piece of furniture of the room.

The Centre or General Table.—This table, (N, Pl. 2,) compactly fitted to serve the double purpose of an operating table for distillations, and other large general operations of the laboratory which would occupy too much room upon the smaller table, Fig. 24, has its top, also of cherry, projecting two inches all around and grooved, glued and tightly jointed, as directed for the preceding table, like which, its lower portions may also be of white pine. Its position is near the centre of

### THE LABORATORY-THE CENTRE-TABLE.

the room, so as to afford free access to all its sides. Fig. 29



gives a view of it. Its dimensions are 2.10 feet height; 6.6 feet length; and 3.4 inches breadth. In order to ensure perfect stability, the legs are fitted to a bed which is to be firmly screwed to the floor, so that the table may be stationary and free from oscillatory motion, as any jarring may create serious damage to a delicately arranged apparatus.

The drawer space should not exceed 15 inches of the whole height of the table. The end drawers are necessarily, from the construction of the table, very short, and may be omitted entirely, though it is better policy to have as many receptacles as possible, for they will all be found useful as well as convenient.

Of the front drawers, one should be appropriated exclusively to the sheets and other articles of India rubber. Accompanying these must also be a pair of shears and a ball of very fine linen twine for fashioning and securing joints. Another drawer must be reserved exclusively for the corks of assorted sizes. Two smaller apartments or divisions are also necessary, one for the rat-tail files of different sizes, and the other for the cork borer, of which more will be said hereafter.

The stock of filtering paper is also kept in another of these drawers; and with it, the circular tins by which it is cut into different sized filters. The shears for cutting the paper should be kept always sharp and clean. Another drawer divided into compartments is required for the reception of tow,

raw-cotton, bladders, string, &c.; and another for the clean dusters and towels of the establishment.

The filtering cloths and material for that purpose are also kept in a separate drawer. The thermometers and hydrometers are likewise kept in a distinct drawer.

There are many other articles which are better preserved in drawers, and hence there is a necessity for the whole number in the table. The short drawers in the end of the table can be reserved for minor matters, such as the scratchingdiamond and similar implements.

The lower bed of the table forms an excellent shelf for the filter stands, retort-holders, clamps, supports, and other wooden apparatus in frequent use upon the operating table.

All the iron stands and similar apparatus should be painted with black varnish\* in order to preserve them from rust. In the selection of iron hollow-ware for purposes of ebullition, or evaporation, choose that which is enameled internally;—it is more convenient, readily cleansed, and not much more costly than the naked iron ware.

The mouth blow-pipe table occupying a position against the front wall, and immediately under the right window, as



\* To fused asphaltum, 40 ozs., add a half gallon of boiled linseed oil, 6 ozs. each of red lead and litharge, 4 ozs. dried and powdered white copperas. Boil for two hours, then mix in 8 ozs. of fused dark amber gum, and a pint of hot linseed oil, and boil again for two hours more. When the mass has thickened, withdraw the heat and thin down with a gallon of turpentine. shown at p Pl. 2, is an indispensable piece of apparatus which will be more fully spoken of under blow-pipe operations.

The blast or pneumatic table (shown in position at q Pl. 2), which is sometimes also called the table blow-pipe, may be considered as an implement indispensable to the chemist, it being alike useful for bending glass tube, blowing bulbs and other small apparatus, and for rapidly effecting the decomposition and ignition of substances, which, for their fusion, would require an ordinary wind furnace. The most convenient form of this apparatus is shown by Fig. 30. The drawing is taken from one, in Professor Booth's laboratory, made by J. Bishop, machinist of this city. It consists of a brass cylinder piston 2, worked by a treadle which drives the air into a large tin box enclosed in a frame-work 1 immediately under the top of the table. From the front end of the box a tube rises through the table top, and terminating with its small jet within the interior of an Argand burner, urges the air directly upwards, producing a full flame. The Argand burner may be connected with a lamp or reservoir, containing a solution of oil of turpentine, or alcohol, or with a

Fig. 31. gas pipe. In the former case, the burner has a circular wick with a contrivance for adjusting its height. The latter, being neater, and always ready, is almost exclusively used in the laboratory, as giving a powerful flame which may be elevated or depressed at pleasure. With one of

the new fashioned Argand gas burners as shown by Fig. 31, this table forms an excellent substitute for ordinary furnace operations.—(Encyclopædia of Chemistry.)

Air-Pump.—The small table, at r Pl. 2, is used for the air-pump which, when not in use, should be kept in an appropriate place in one of the cases in the balance room. Being a costly apparatus, it is now almost exclusively replaced by syringes, which are more economical and not much less convenient, as made for the purpose at the present time. For the sake of a convenient uniformity, the attachment screws should have a thread similar to that of the stopcock, so as to admit of a ready adaptation to each other when an attachment is to be effected. Of the many operations in which the syringe is made to assist, may be mentioned the displacement of air in retorts, globes, and other vessels, &c., previous to the introduction of gases, and also in



the exhaustion of receivers for experiments with atmospheres of less than ordinary pressures.

In order that these machines may work properly, it is necessary that the joints should be tight and free from leakage, and that the pistons be well oiled so as to promote their easy motion. An excellent method of preserving this apparatus in good order, is to work it frequently even when not in use, for by this mode, the elasticity of the pistons and ready play of the cocks may be retained. Fig. 32 represents a horizontal

Fig. 32.



double cylinder air-pump, invented by A. L. Kennedy, M. D., of this city. The advantages of this apparatus over the old form, are stability and portability, and greater cheapness. Besides, it is more easily and readily worked. Unlike the upright cylinders, this pump is not liable to tilt over whilst being worked, and consequently there is not that instability so annoying when using the barometer gauge.

"In the figure, L, L represent the barrels, the enlarged ends of which are let into the board and bolted through to insure stability. There is one rack; the two pistons being attached to its extremities. A portion of the rack is exposed at T. The semi pinion w, works in cast straps, or gudgeons, attached to the bottom of the board by screws, which, passing through, terminate in the rack guides, one of which is seen above. The forward gudgeon is so cast as to receive the end of the clamp which secures the pump to the table. The semi-pinion

### THE LABORATORY-THE AIR-PUMP.

works upwards through a slot cut in the board, and of course between the rack guides. The upper extremities of the guides are perforated to receive rollers, against which the back of the rack may work when necessary. None have yet been required. To the axis of the semi-pinion the handle is attached in the usual manner. The piston may be either solid or valved, and the cylinders may communicate with the plates R and B, in the way most approved by the maker. In the pump from which the sketch is taken, the pistons are solid. The farther extremities of the cylinders bear female screws, which connect with corresponding male screws on the block. On the posterior portion of each block is cut a female screw; the male of which bears the valve, of course opening inwards, v, v. On those portions of the blocks which project into the board are cut male screws bearing valves opening outwards. Perforated nuts over these secure the blocks to the board, and the valves against injury. At v, v is attached the tube leading from the plates. D is the screw for restoring atmospheric pressure. The general stop-cock s, connects this with the parallel tube which, bearing the gauge cock s', forms at pleasure a communication between the plates.

The original of the figure both exhausts and condenses. The remaining letters refer to the parts used in condensing. This is effected by simply connecting, by means of tubes under the board, the valves F', F with a third tube passing upward to the stop-cock K. Then the air drawn in at R, will be condensed in a receiver screwed on c. Those familiar with Pneumatic chemistry need not be told of the facilities thus afforded for the transfer of gases. The condensing gauge is borne by the screw G. To the practical chemist, it is unnecessary to dilate upon the advantages that result from lowering the centre of motion to a level with the points of support, bringing *both* plates directly under the operator's eye, and presenting, at about the cost of an ordinary exhausting pump, an instrument furnished with all the facilities for exhaustion, transfer and condensation, without any shifting of parts."

The table s Pl. 2, to the right of the air-pump, is a stand for the common scales of the laboratory, which are useful for testing the weights of materials purchased and for weighing coarser articles in large quantities. A cheap platform balance with a movable tin dish answers conveniently for this purpose. The accompanying (Avoirdupois) set of weights should range from  $\frac{1}{4}$  oz. to 8 lbs.

# THE LABORATORY-THE CUPBOARDS.

The next fixtures to be described are the cupboards. Those affixed to the partition of the furnace room as at t and u Pl. 2 are more properly shelves, with curtains instead of doors to protect their contents from the dust. The set t may be occupied with the leaden coils, wooden and coarser apparatus of the apartment. The curtains of common muslin, rendered fire proof by immersion in a solution of borax and sal ammoniac and drying, are hung by means of small brass rings upon an iron rod running the whole length of the cap of the shelving; and in order to keep them distended, leaden bullets should be sewed at occasional distances upon the lower ends. The shelving u ascends to only half the height of that of tbecause the upper space is to be reserved for racks and rings. The shelves are intended as receptacles for the porcelain capsules, crucibles, &c., the bell, beaker and other similar glass apparatus, always taking care to occupy the lower shelves with the larger and heavier articles. The tube rack is nothing more than a series of pegs, placed closely adjoining in a straight line and inclining upwards so as to prevent the tubes from falling through. This open work presents the whole stock of tubing to view at one glance, and enables a ready selection of any particular piece of rod or tube. The smaller pieces which would be apt to fall through, should be kept in a drawer of the centre table specially appropriated for the The remaining portion of the upper space must be purpose. furnished with a series of various sized spikes to hold retort and flask rings. These rings, readily made of wire, vary in size from a half to two or more inches in diameter, and receiving the necks of retorts and other curved or bent apparatus, retain them in a safe and convenient position. The rings may also occupy any small vacancies upon the walls for the use of such a portion of the apparatus as the cupboard cannot contain.

The small cupboard (v Pl. 2) in the corner opposite, may be used as a sort of general cupboard for very nice little matters, which require great care and cleanliness in their preservation. The door consequently should be fitted with a fastening and kept constantly closed when not in use.

The cupboards w and x erected against the partition opposite, and occupying the spaces on either side of the entrance into the office are, the one x for the stock of drugs and chemicals; the other w for the new empty bottles, to be confined to the lower shelves, and for the specimens that may from time to time be accumulated by the labors of the operator. The lower half of the cupboard X should be furnished with small drawers similar to a druggist's case. These are for the dye woods, sulphur, chalk, and other similar coarse articles of stock which are more securely kept in this way than in bundles, which are liable to rupture and damage by rough handling and by retaining moisture. The upper shelving is to be exclusively occupied with the articles in bottles, which are to be arranged in groups, the compounds of each base forming a group. The mineral and vegetable acids and organic compounds, have also each a separate position. The weightier articles as elsewhere directed, should always occupy the lower shelves, both for convenience of handling and on account of their greater stability and power of bearing heavier weights than the upper shelves.

The black board y Pl. 2, is hung sash-like between the uprights of the cupboard w and x and, being counterbalanced by weights, can be lowered or raised at will, and thus presents no hindrance to egress or ingress from and to the office. For rough calculations and plans, drafts of apparatus, diagrams, &c., the black board is very convenient. When a hand slate is substituted, the pencil should be of talc (French chalk) which makes a more distinct mark than the common slate pencil, and gives more facility in writing. These pencils are now sold in most of the stationery stores.

Bottles.—Particular regard must be had to the shape and material of bottles for laboratory use. Those intended for holding acids or salt solutions, must be of well annealed glass, which is free from lead and can resist the corrosive action of their contents. Some glasses containing an excess of alkali, gradually lose their brilliancy by absorption of moisture from the atmosphere; others again are attacked by acid and alkaline solutions; and some indeed, even by prolonged contact with boiling water.

The inalterability of glass by air or chemical agents (hydrofluoric acid excepted) is proportional to its hardness and infusibility. Flint glass is the most brilliant and comparatively fusible, and its consequent pliability renders it available for thermometer and barometer tubes, but as material for chemical vessels it is far inferior to the Bohemian glass (a silicate of potassa and lime with large traces of alumina), which is harder, lighter, and while possessing many better qualities for chemical ware is, when well made, scarcely less remarkable for beauty than crystal lead glass.

Care must be taken in the selection of glass apparatus, especially those which are to serve as implements for reactions, to choose such as are free as possible from striæ, knots, or bubbles, defects owing to the imperfect mixture of the materials of the glass. The more transparent the glass, the more readily can the interior cleanliness of the vessel be ascertained. The common green glass bottle from the factories of New Jersey, in the absence of better, answers every purpose for the common acids, coarser dry substances, and the solutions of such as are soluble; and are, moreover, economical. For the reagents and finer chemicals, there is a cheap white glass, free from lead, manufactured at Storms and Fox's Factory in Kensington, Philadelphia, which is well adapted to the purposes, and replaces sufficiently the elegant, but at the same time much more costly Bohemian glass which is only to be obtained by importation. The laboratory series should vary in size from one ounce to one gallon, ranging as follows, 1, 2, 4, 8, 16, 32, 64, 128 ounces. The most approved shapes are shown by the cuts below. Fig. 33 represents a



wide mouth bottle for powders and crystals. It is short and wide, with round shoulders to admit of ready emptying and cleansing, and has a strong tall neck for tightly corking. The corks should be perfectly smooth and of the velvet kind. This shape is equally applicable to the bottles of white glass, as is also that of the narrow mouth, glass stoppered, as shown by Fig.

35. The narrow necks and their stopples, must be accurately ground so as to insure perfect tightness. As the cost of this white glass above mentioned is so very little greater than the Jersey green, it would probably be more advisable to purchase the whole suite of bottles of such material. The stopples of the narrow-necked bottles are made nearly spherical, but

# THE LABORATORY-THE BOTTLES.

somewhat flattened on the top to project over the mouth so as to protect it from dust. The lips are flat and stout for pouring readily. The wide mouthed stoppered bottles are, as to body, similar in shape to the above, but their stopple-heads are flattened and cover both the mouth and the rim. The series of all these bottles consists of the sizes above mentioned. For one or two substances both in solid and solution, which are sensitive to the decomposing influence of the light, nitrate of silver and protochloride of mercury for instance, it is necessary that the bottles be either of dark colored glass or else covered exteriorly with tin foil. For hydrofluoric acid a lead bottle is necessary, as glass is decomposed by that body. All solutions should be kept in ground stoppered bottles, and if economy is indispensable, let the series consist of as many of the green glass bottles as possible, retaining only as many of the white Bohemian glass as are absolutely necessary for the finer reagents. Corked bottles are inconvenient and liable to leakage, and their use as permanent receptacles of liquid should, if possible, be entirely discarded. We have consequently not given the shape of a narrow-mouthed unstoppered bottle, though if they must be had, the shape of Fig. 34 with the neck narrowed must be the pattern.

All bottles with contents must be labeled in full and with symbols. This injunction as to labeling applies with equal force to the beaker glass upon the sand-bath and the capsule over the lamp, and to every vessel resting upon the shelves or employed in operations, which contain any substance or solid, whether the material or product of any process. An omission of this precaution frequently leads to much confusion, and occasionally to serious errors. Thin writing paper glazed upon one side with a solution of gum tragacanth, and divided into small squares of different dimensions to suit the several sizes of vessels, answers very well for the purpose of labeling operating vessels. With a pencil, or more properly pen and ink, the designation may be written on the label, and thus completed, is to be pasted on the bottle. For bottles containing the chemicals, materials, &c., these paper squares are equally applicable, but for the test series upon which the labels are to be permanent, it is better that the names be etched upon the glass by the action of fluohydric acid. In England they manufacture a bottle for this purpose, with indelible names in black enamel, upon a white ground. They are, how-

# THE LABORATORY-CLEANSING OF GLASSWARE.

ever, costly. As a substitute for either of the two latter, are printed labels after the patterns of those published by La Rue & Co. (110 Bunhill row, London), which contain the full name of the articles, its symbol, and equivalent. Those bottles of the test series, which are to contain the acids or other corrosive liquids, wholly or in part volatile, should be provided with ground glass caps. Fig. 36 represents a bottle of this

pattern with the label corroded in by fluohydric acid. Fig. 36. Nitric

The mouths of all the test bottles should flange in order to facilitate pouring. The last drop of liquid generally adhering to the lip can be arrested by touching it with the stopple, which catches and re-conveys it to the bottle when returned to the mouth. Let it therefore be a cardinal rule of the laboratory, that no experiment or operation shall be abandoned even for a moment without having the receptacle labeled.

There are other laboratory uses, independent of the aforementioned, to which bottles are applicable. The widemouthed when accurately stoppered and rendered air tight in the mouth with a little lard or suet, are excellent substitutes for jars, for the reception and safe keeping of gases which are soluble in water or corrosive of mercury, and consequently cannot be collected over either.

Cleansing of Glassware.-When bottles or glassware are greasy, the aid of alkali or ashes is necessary for its removal. In open vessels bran or saw-dust, by their mechanical action, will cleanse the surface of grease. In either case hot water

Fig. 37.



is a great assistant. An iron or copper kettle, Fig. 37, fitted to the top of the stove or one of the openings in the top of the furnace, is a convenient vessel for furnishing a constant supply. The rinsing afterwards may be with cold water. A short twine brush, similar to that used by housewives for washing tea, things, is an excellent assistant in cleansing operations, and there should be several of them

about the laboratory. For alkali, lime as an example, which coats the sides, a little common muriatic acid is requisite. When the dirty matter is fixed and resists the purifying action

# THE LABORATORY-CLEANSING OF GLASSWARE.

of these two agents, and also of hot water, resort must be had to the use of shot, which, when agitated with a little water in the interior of the bottle, gradually removes the adherent dirt, which can then be rinsed out with clean water. Carelessness in leaving behind one or more shot, which frequently secrete themselves in the crease at the bottom, may result in injury to the next contents of the bottle, if it be solvent of metal. Coarse sand and angular pebbles, which are sometimes substituted for shot, are apt to scratch the glass, a disadvantage which does not apply to small round pebbles. The daily ablution of apparatus had better be performed at the close, and after the labors of the day, so that the advantage of the night may be obtained for draining and drying. torts and beaked vessels should be ranged on shelves with circular holes for the reception of their beaks. In this case as well also in that of open vessels, the mouths should always be placed downwards. When it is necessary to dry the cleansed vessel for immediate use, it may be well wiped with a towel exteriorly and then placed upon a moderately heated sand bath, which will soon expel all internal moisture. Wide mouthed vessels can be dried with a cloth. For cleansing test tubes, a goose-feather or stick with a small sponge fastened to its lower end is very convenient.

The removal of corks from the interior of bottles is effected by an instrument consisting of four strands of iron wire, of about one foot length each, united together at one end, and at the other four extremities bent into an angular shape. Being elastic, there is no impediment to its passage through the mouth of the bottle, in the interior of which it is made, by a dexterous management, to catch and secure the cork, which can then be drawn out with the wire. This simple little instrument is to be purchased at any house-furnishing bazaar. A very convenient substitute is a doubled string; the loop thus formed, when introduced into the bottle, secures the cork and allows its easy extraction.

It not unfrequently happens with ground-stoppered bottles, in cases where certain substances form their contents, that the stopple adheres so firmly as to resist all efforts to remove it with the fingers. It is then necessary to tap it gently and alternately on each side with the handle of a spatula,—the spatula being held by the blade, and the bottle, by the top of its stopple—the body resting on the table, in the other

### THE LABORATORY-THE TEST-CASE.

hand. In ordinary cases this process loosens the stopper, but if it fails, it then becomes necessary to carefully expand the neck over the flame of the small spirit lamp, and in order that it may be uniform, the bottle must be kept constantly revolving in a horizontal position. When sufficient warmth has been applied, a gentle tapping of the stopple, as above directed, effects its removal. After the neck of the bottle has cooled, it and the stopper must be washed and dried before the latter is returned to its place, otherwise it will soon become tightened again. The plan sometimes adapted of inserting the head of the stopper in a chink and then wrenching it out as it were by turning the bottle with the hand, is not advisable, as it endangers the safety of both the vessel and hand.

When the lamp is used, the motions must be dexterous and careful, so as to confine the heat to the neck of the bottle, for if it is allowed to reach the stopper also, the expansion of both being then equal, the removal of the former cannot be effected. The success of the effort depends upon a difference of temperature between the stopple and the neck which encloses it. Friction, induced by drawing a string constantly, and for a length of time, to and fro around the neck of the bottle, is sometimes substituted for the heat of a lamp.

When the cementing matter is a crystallized salt, hot water placed around the edges will loosen the stopper by dissolving the salt;—when it is metallic matter, hydrochloric acid is necessary, care being requisite that it does not injure the contents of the bottle. In some cases olive oil, similarly applied, is more effectual than either hot water or acid.

These remarks are equally applicable to nearly all kinds of closed glass vessels. Broken glass and odd stoppers being often needed for various uses, should be preserved in a box for the purpose.

The Test-case.—The bottles of the test-case should be of white glass, entirely free from lead, and nicely fitted with ground stoppers. As they are constantly in use, it is preferable to etch their labels upon the glass. This is readily done by the operator himself, who has only to coat a limited space of the bottle (see Fig. 36) with melted wax, and after tracing thereon, with an iron style, the name and symbol of the reagents to be contained therein, to wet the marks with sulphuric acid, and then sprinkle on some finely powdered fluoride of calcium (fluor spar). The fluohydric acid thus set free attacks the glass,

# THE LABORATORY-THE TEST SERIES.

and renders the latter opaque and distinct, whilst the wax protects the other portion from its action, and when removed, presents the smooth surface of the glass. Care should be taken to avoid contact with any of the escaping vapor, as it is deleterious.

When paper labels are used they must be payed over with a thick coating of insoluble varnish, and written upon with incorrodible ink. The former consists of white of egg (strained), which is to be applied with a camel's hair pencil, and immediately coagulated by steam heat and then dried in an oven at about 212° F. The ink is made by dissolving one part of genuine asphaltum in four parts of oil of turpentine, and adding lamp black to render it properly consistent. The neatest method of marking the labels with the ink is by means of a small stamp and types. When the ink has dried, the varnish is to be applied as above, but preferably after the label has been pasted (with gum tragacanth) upon the bottle. The transparent film hardened, and rendered insoluble by heat, presents a firm resistance to strong acids, alkaline solutions and other reagents, and, moreover, this kind of label is economical.

The test series consists of eighty-two bottles, which have their position in the case over the operating table, Fig. 24. Of this series, there are eighteen narrow-mouthed pints with contents as follows:

1	Sulphurous acid (in solution)	SO.
2	Hydrochloric acid (common)	HCI
3	" " (pure)	HCl
4	Chlorine water (in solution)	Cl+H
5	Nitrie acid (common)	NO <sub>5</sub>
6	" " (pure)	NO <sub>5</sub>
7	Sulphuric acid (common)	SO3
8	" " (pure)	SO <sub>3</sub>
9	Nitromuriatic acid (aqua regia)	NO <sub>4</sub> +Cl,HO
10	Hydrate of potassa (in solution)	ко+но
11	Aqua ammonia	NH <sub>4</sub> O
12	Carbonate potassa (in solution)	KO,CO <sub>2</sub>
13	" soda " "	NO,CO <sub>2</sub>
14	" ammoniæ "	NH <sub>4</sub> O,CO
15	Acetate of lead " "	PbO,A
16	Sulphate of lime " "	CaO,SO,
17	Lime water " "	CaO+HŎ
18	Sulphuretted hydrogen "	HS

The next size (narrow-mouthed) is eight ounces, and of these there are nineteen with liquid contents, as follows:

# THE LABORATORY.

19	Acetic acid	C.H.O. or A
20	Oxalic "	CO, H
21	Tartaric "	$C_{o}H_{i}O_{i}=\overline{T}$
22	Phosphorous acid	° PO3
23	Ether	C,H,O
24	Chloride of ammonium	NHCI
25	Hydrosulphuret of ammonia	NH <sub>4</sub> S <sup>+</sup> HS
26	Oxalate of ammonia	NH <sub>4</sub> 0,0
27	Chloride barium	BaCi
28	Chloride calcium	CaCl
29	Phosphate soda	HO,2NaO,PO
30	Sulphate copper	ĆuO,SO <sub>s</sub>
31	Basic acetate of lead	3PbO,A
32	Proto-sulphate of iron	FeO,SO,
33	Sesqui-chloride of iron	Fe, Cl
34	Sulphate of magnesia	MgO,SO, HO
35	Sulphuret of Potassium	KS <sub>5</sub>
36	Sulphate of alumina	Al <sub>2</sub> O <sub>3</sub> ,3SO <sub>2</sub>
37	Infusion of galls	2 3' 3
The	form own one much on ton of	high the limit of

The four ounces number ten, of which the liquid contents are as follows:

38 Bitartrate of potassa	$KO,HO,\overline{T}$
39 Acetate ""	KO,A
40 Basic silicate "	3KO,Si <sub>3</sub>
41 Chloride of mercury	Hg,Cl,
42 Proto-chloride of tin	Sn,Cl
43 Proto-nitrate of mercury	Hg <sub>g</sub> O,NO <sub>5</sub>
44 Chromate of potassa	KaO,CrO <sub>3</sub>
45 Sulphate of potassa	KaO,SO3
46 Succinate of ammonia	$NH_{0.S} = (C_{1}H_{0.S})$
47 Borate of soda	NaO,2BO3

The two ounces, eight in number, contain of liquids as follows:

48	Bicarbonate potassa	KO,2CO2
49	Acetate of baryta	$BaO,\overline{A}$
50	Ferrocyanide of potassium	2KCfy
51	Ferricyanide of potassium	K <sub>3</sub> ,Cfy <sub>2</sub>
52	Baryta water	BaO+HO
53	Nitrate of silver	AgO,NO5
54	Iodide of potassium	KI
55	Solution of indigo	

The liquid contents of the one ounce test bottles are,

- 56 Carbazotic (nitropieric) acid
- 57 Nitrate of nickel
- 58 Proto-nitrate of cobalt
- 59 Nitrate of potassa
- 60 Ammonio-nitrate of silver

 $\substack{ {\rm C_{12}H_3,N_3O_{13}+Aq}\\ {\rm NiO,NO_5}\\ {\rm CO,NO_5}\\ {\rm KO,NO_5}\\ {\rm AgO,NO_5+2NH_3} }$ 

# THE LABORATORY-THE TEST SERIES.

61	Bichloride of platinum	Pt,Cl.
62	Chloride of gold	Au,Ci
63	Caustic, soda	Na,O
64	Antimoniate of potassa	KO,SbO,
65	Cyanide of mercury	· Hg,Cy <sub>2</sub>

In addition to the narrow-mouthed, there are required fifteen wide-mouthed glass stoppered bottles. The contents of these are as follows:

In the eight ounces

66 Mixture of carbonates of soda and potassa	NaO,CO,+KO,CO,
67 Carbonate of lime	CaO.CO.
68 Sulphuret of iron	FeS
69 Dry carbonate of soda	NaO,CO, (dry)
70 Carbonate of baryta	BaO,CÒ,
71 Cyanide of potassium	KCy "
72 Granulated zinc	Zn
73 Per-oxide of mercury	HgO,
In the four ounces;	
74 Hydrated oxide of bismuth	BiO,+HO
75 Oxide of lead	PbO
76 Blue litmus paper	
77 Red " "	
78 Turmeric "	
79 Georgina "	
QO I and " "	

81 Starch paste

A leaden bottle of two ounces capacity, for the hydrofluosilicic acid 3HF, +2SiF<sub>3</sub> completes the series.

All these bottles should be made heavy, for if too thin, being so frequently handled, they are liable to be broken. Of the preceding numbers, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 18, 23, 25, should be furnished with ground glass caps as shown by Fig. 36; No. 53 must be of dark glass or else covered exteriorly with tin foil. Nos. 77, 78, 79, 80, 81, should always be accompanied with a pair of pincers with platinum points similar to those used in blowpipe operations, as the test papers should never be handled with the fingers. The bottles for alcohol (C4H6O2), and distilled water (HO) may be of common green glass, narrow-mouthed and quart sized. They are fitted with double tubes so as to insure a gradual egress of the liquid; and are designed as conveniences to the operating table, for supplying small quantities of their contents to test tubes and narrow-mouthed vessels without the aid of a funnel. Fig. 38 shows their form and arrangement.

### THE LABORATORY.

A piece of bright copper and one also of iron are also frequently needed as reagents.

All of the forenamed reagents must be chemically pure, as also the water used in making solutions of them. The processes by which they are prepared, would not be altogether inappropriate to this work, but more pertinent matter demands our space and so we refer the operator to an excellent treatise upon the subject, by Mr. E. N. Kent, practical chemist of New York, and now in the progress of preparation for the press.

Besides these reagents, a small stock of which should always be kept in reserve on the shelves of the cupboard, there is required a general assortment of drugs and chemicals in limited quantity. The coarser and cheaper articles of this stock should preferably be purchased from the dealers, but it is advisable for the operator to prepare the costlier ones for himself, not only on the score of economy, but also because of the practice which he will acquire in the manipulations of various processes.

There remain but few points to be remarked upon before closing our chapters upon the laboratory. We have already enjoined upon the experimenter, great cleanliness, and we now repeat the injunction. The hands should always be free from dirt, and invariably washed with castile or palm soap before going to meals. This precaution is absolutely necessary on account of health, for otherwise, in working with deleterious matters, the little particles which secrete themselves under and around the finger nails, may be conveyed into the system and thereto work an injury. So also, when engaged at one time upon several operations of a different nature, it is necessary to rinse the hands in passing from the management of one to that of another of them. For this purpose, the hydrant or reservoir with its adjoining handtowel, Fig. 22, is very convenient.

To protect the person from dirt, the operator should provide himself with a suitable costume. A long wrapper of linsey or baize for winter, and of Holland linen for summer, is very suitable. A light cap of some cheap material, is a good shield to the hair against the bad effects of dust and vapor.

In all investigations, the practice of working upon small

### THE LABORATORY-RECORD OF ANALYSES.

quantities, will lead to habits of nice and delicate manipulation. Besides, it is easier, less costly and fatiguing to manage a small portion of any substance. Record your processes in the *laboratory book*, to be kept specially for the purpose; note in detail the modus operandi pursued, and the results with the day and date, so that you may have every facility of drawing a clear conclusion from the results of your labors.

There are two other books needed, one is the Record of Analyses, in which are transcribed the analyses of such substances as may undergo examination in the laboratory. Their mode of analysis may also be annexed. This record is very useful for future reference. The other book is an "Index rerum" after the plan of the Rev. J. Todd, author of the Student's Manual. As it is impossible to retain in the memory all that one reads or sees in the numerous works which come under his eye; and as we meet with much that is valuable. and really worth preserving, we must resort to some other means more practicable and less laborious than copying out extracts. Mr. Todd recommends the habit of making an index rerum of reading. This book consists of several quires of blank sheets, letter form, and is alphabetically classified, so as to exhibit at a glance, the name of the book and the number of the page treating of the subject, the synopsis of which is recorded under its appropriate letter and heading. There are many facts and opinions met in reading, especially in the journals, which are certain to be wanted some day or other, and by thus gradually storing up you save yourself a deal of trouble for the future when there is need of research upon any subject; and in a few years will have accumulated a mass of information of incalculable value in the practice of your profession. Always, as Mr. Todd directs, have your index at hand when reading book, journal, pamphlet, or newspaper; and "when you meet with anything of interest, note it down, the subject, the book, the volume, and the page; and make your index according to subjects as much as possible, selecting that word for the margin which conveys the best idea of the subject," so that there may be no difficulty in finding the original place when it is necessary to refer to it. For example, in reading the journals for this year, you find several articles which you may wish to refer to again, and so note down their subject matter as follows:

#### THE LABORATORY-INDEX RERUM.

# A ACIDS FATTY.

Their constitution, new theory of, by Jas. C. Booth, Journal of the Franklin Institute for 1848.

Different views of its composition. Encyclopædia of Chemistry, p. 488.

G

GUN COTTON.

# S

# SUGAR.

Analyses of, by circular polarized light, Report to Congress by Professor R. S. M'Culloh, 1847.

There are many other minutiæ that might be mentioned, but for want of room for more important matter; they will, however, suggest themselves in the progress of operations.

Habits of industry, close observation and neatness, are indispensable to the acquisition of a proficiency in manipulation, without which it will be difficult to form correct conclusions.

The laboratory which we have described, is well appointed for every branch of research. Many of the implements enumerated may be dispensed with for ordinary operations, but they are requisite for a complete arrangement, which, as given in the preceding chapters, is not at all extravagant. Moreover, with a little extra industry, the operator can soon realize the outlay for all conveniences, in the manufacture and sale of such pure chemicals as may be in demand. We have provided him with every appliance for the purpose, so that his self improvement may be attended also with pecuniary profit.

Where the means are limited, it is better that the purchase of apparatus should be gradual, commencing with those pieces which are of most general use. This course judiciously carried out, will in time possess the owner of a well stored laboratory. All stock and apparatus can be bought from the manufacturer, or importer, at very little over one-half the dealer's prices, for the same articles.

# CHAPTER IV.

### DIVISION OF SUBSTANCES.

THIS operation is a mechanical process, by which the surface and points of contact of solid bodies are multiplied; thus diminishing, in a high degree, the opposing force of cohesion; and, consequently, by promoting greater access to its particles, enabling the more ready and rapid action of reagents upon solid matter.

The means by which the division of solid matters is accomplished are manifold, and vary with the nature of the substance to be reduced; some bodies being pulverizable by almost any of the processes, while others again require a particular method for their reduction. The different modes of operating may be classified as follows:—

1st. *Slicing.*—This process applies to fibrous matters, and is practised with a lever-knife, similar to that used by tobacconists for cutting tobacco, and shown by Fig. 39.



Being thus reduced to thin slices, the substance is in better form for maceration, &c.; and, moreover, admits of readier desiccation, a necessary process when it is required to be further reduced under the pestle, or by being grated on a coarse rasp.

This mode of pulverization by rasping, though particularly applicable to fibrous substances, such as fresh roots and the like, is sometimes used for metals and hard matters. In the latter case, the files must have finer and sharper teeth, and in both instances be perfectly clean, and free from grease and dust.

2d. Contusion.—In order to attain a minute division of the denser substances, whose particles are very cohesive, resort must be had to the pestle and mortar. The material of this ap-

paratus varies with the nature of the substance to be powdered. To prevent errors, corrosive or caustic matter should never be pulverized in metallic mortars, else by a solution of a portion, or contamination with abraded particles, unavoidable confusion will ensue. The resistant nature of the material of the mortar must be proportional to the hardness of the body to be operated upon. For the harder insoluble substances, those of iron, brass, or steel are generally used. For the less dense and more pulverizable bodies, especially those which are acid, or corrosive, porcelain, wedge-wood or glass is the proper material. Marble, being readily attacked by acids, mortars of that material are only used for reducing those inert substances which are readily comminuted merely by trituration, such as chalk, neutral salts, &c. This material, as well as glass, is well replaced by porcelain or wedge-wood, which are stronger, and otherwise much less objectionable. There should be an assorted series of mortars for laboratory purposes.

The large iron mortar has its position in the furnace-room, and is permanently and firmly fitted upon a block, in some convenient place, for general use, in pounding ores, metals, and coarser substances. The pestle of this, as of all other mortars, should invariably be of one piece and of the same material as the mortar; because, when the lower part is fitted to a handle, it is apt to become loosened and drop off particles of the cement with which it is fastened, to the injury of the contents of the mortar. The handle or upper portion must afford convenient space for grasping, and the base or lower portion, roughened on its face by use of sand, should diverge to a diameter of about one-fourth of that of the mouth of the mortar. Fig. 40 exhibits a mortar of proper form and pro-



portionate thickness as to its different parts. Its interior form is nearly that of the butt end of an egg, so as to promote a constant contact of the matters being contused with the rotating pestle. To prevent the ejection of particles of matter and the escape of dust, and consequent inconvenience to the operator, as the case may be, the mortar should be provided with a sheep skin conical coverlet, with a hole in its centre for the passage of the pestle, which is to be fastened around its rim and over its mouth, with a string. Circular pasteboard and wooden covers, of sizes corresponding with the mortars and with a hole in their centres, are often substituted for the conical coverlet. The operator should always stand with his back to a current of air; and to further guard against the unpleasant or deleterious effects of the fine dusty particles which may arise from the mortar, he can moisten its contents with a little water, provided that liquid is without action upon the substance. Exposure to warmth, for the evaporation of the water, will restore the matter to its original dryness.

All substances formed of an organic tissue should be previously dried, so as to afford greater facility in their pulverization. A previous reduction of ores, and coarse hard substances into lump, by concussion with a hammer upon an anvil, and of roots and the like into slices or bits with a common knife or lever cutter, (Fig. 39,) are preliminary processes which greatly facilitate their pulverization. The substance to be struck upon the anvil should be previously wrapped in strong brown paper.

Silicious stones pulverize much more readily after having been heated to redness in a crucible, and in that state projected into cold water. This increased friability is occasioned by the unequal cooling of the mass.

Metals, alloys and the like, which are difficultly pulverizable whilst cold, may also be readily crushed when heated to redness.

When it is required to reduce the substance into small fragments only, it can be broken down by a succession of blows with the pestle. If the substance is very hard, the force of the arm should be added to the descending weight of the pestle, so as to impart power to the blow. A subsequent circular grinding motion of the pestle, continued for a length of time will further reduce these fragments to fine powder, and consequently this movement must be avoided when only a coarse comminution is desired. The mortar must always rest upon a solid foundation, and during the operation of pounding should be occasionally shaken, in order that the coarser particles which mount to the sides may be forced back to the centre of the mortar; so as to receive the full effects of the descending pestle, which should never be allowed to strike the sides of the mortar. If the substance is to be reduced to a fine powder, the process is greatly facilitated by operating

### DIVISION-MORTARS.

upon only a small portion at a time, as the pestle is less liable to become clogged.

In the analysis of rare minerals, especially those which are very hard, the reduction is effected in a small mortar of hardened steel. This apparatus, shown by Fig. 41, consists



of three separable pieces, each of which is smoothly turned, so as to present an even surface exteriorly and interiorly. Cis the base piece into the cavity of which the cylinder B fits somewhat loosely. It is this cylinder which receives the mineral to be reduced. Sliding into it is the exactly fitting pestle A, which being struck successively with a hammer, crushes the mineral to powder without waste of any of its particles by ejection.

When the powder thus obtained is not yet sufficiently fine for analysis, it must be transferred to an agate mortar, and rubbed with the pestle until reduced to an impalpable state. The pestle and mortar are of the same material, the hardness and smoothness of which render it particularly applicable for the purpose. The motion of the pestle should always be circular, otherwise a perpendicular blow may endanger the safety of the mortar, especially if it has a fissure, as is often the case, running through it. The given weight of the mineral for analysis must always be estimated after pulverization; never previously, lest a loss by ejection, or adhesion to the mortar, or spatula may lead to inexact results. Fig. 42 ex-

#### TRITURATION-PORPHYRIZATION.

hibits an agate mortar, which can be purchased of sizes varying from 1 to 6 inches in diameter. One of about  $3\frac{1}{2}$  inches width will be most useful. It should be selected as free from indentations, fissures or cavities as possible, for these faults not only impair the durability of the mortar, but render its cleansing very difficult. An excellent

Fig. 42.



plan of removing tenaceously adhering matter from the sides or bottom of a mortar, is to rub them with pumice stone and water.

3d. *Trituration.*—This mode of manipulating with the pestle is applicable to those substances which are friable, and fall to powder by being merely rubbed up by a circular or grinding motion of the pestle, and which would soften and become obstinate by being pounded. Chalk and the like, and most of the salts are in the first category;—the resins and gum resins in the second.

Sand is added to facilitate the reduction of resins and similar substances, which cake under the pestle, only when they are intended for maceration or solution. Under other circumstances, the medium would be an adulterant on account of the impossibility of separating it.

The proper material for a mortar for this purpose is white wedge-wood, of form, as shown by Fig. 43. Berlin porcelain mortars, glazed outside and biscuit internally, with broad butted, solid pestles, as shown at Fig. 44, are neat and convenient



implements, but less available for general purposes than those of wedge-wood, which are stronger, more durable, and will stand harder blows. These are purchased by the diametral inch, and the most convenient size is 6 to 8 inches width at the mouth. It will be well also to have a smaller one of the same material, say of 2 inches diameter at the top.

4th. *Porphyrization.*—This mode of pulverization, only employed when it is desired to give the comminuted substance the

greatest fineness, takes its name from that of the material of the vessels in which it is practised. A small porphyry mortar, hemispherical interiorly, or preferably a slab and muller is the apparatus employed. Flint and even glass, which are equally as hard as porphyry, form an economical substitute for that material. It is highly important that the material of the apparatus shall be less easily abraded than the substance being ground; for if too soft, the latter becomes contaminated with the particles which are rubbed off, and, hence, in exact investigations, inaccuracy is caused.

Porphyrization is generally effected by rubbing the coarse powder between a flat slab and muller, until reduced to an impalpable state. The circular motion of the muller disperses the powder over the slab, rendering it frequently necessary to collect it together in the centre with a spatula, so as to keep it uniformly under the action of the muller. The spatula may be of horn or steel, but is better when of platinum. Fig. 45



exhibits a slab and muller. When the substance under operation is unalterable by water, it may be moistened with that liquid, which, by converting it into a paste, facilitates its reduction and prevents any waste by the escape of dusty particles. The powdered paste is easily

dried by being dropped in dots upon a porcelain plate and exposed to warmth. Those matters which are soluble in or alterable by water, must be porphyrized in a dry state.

5th. Sifting .- The impossibility of reducing the whole of a substance at once to a uniform state of fineness by any of the preceding processes, renders necessary an occasional separation, during the progress of pulverization, of the more comminuted portions from the grosser particles. This is effected by means of a sieve, of which there should be several in the A wooden cylinder of about four inches depth, laboratory. with an accompanying ring of the same materials, constitutes the frame, over which can be stretched a cloth of any required fineness. For coarser articles, fine brass wire is the best material for the cloth, but when the powder is to be impalpable, bolting cloth (raw silk), or gauze is requisite. Sieves are also covered with hair-cloth, buckram, book-muslin, and iron wire of different sized meshes, each of which has its appropriate application. The metallic sieves should have their

cloths permanently fitted to them. For all the rest, two frames, as above described, one of much larger dimensions than the other, will serve; as it is only necessary to remove the ring when it is desired to substitute one kind of covering for another. The sieves of cloth, of graduated fineness, can be kept in some secure place, and withdrawn as wanted, and thus we have the economical means of possessing a full suite of sieves from the metallic wire, through all the grades of fineness up to the closest wrought bolting-cloth. The form of a sieve is shown at A, Fig. 46. After the separation of

the finer portions by the sieve, the coarser particles are again subjected to grinding and sieving as often as is necessary to convert the whole into the requisite state of uniform fineness. Horn scoops or porcelain spoons or ladles are the proper implements for transferring the contents of the mortar to the sieve. In some cases a stiff pasteboard card, being more pliable, is a convenient substitute. The use

of the hand, for this purpose, should always be avoided as a slovenly practice. A platinum, horn or bone, or, less preferably, steel spatula may be used to detach the particles adherent to the sides of the mortar. To prevent inconvenience or injury to the operator, (who, both in powdering and sieving, should always stand with his back to a current of air,) from particles of dust or acrid poisonous matter, as well also to avoid waste, the sieve should be fitted with a top and bottom covering, as shown at B and C, in Fig. 46, the upper of which arrests the escape of the light dust into the air and the lower receives that portion which passes through the cloth. These covers are headed with parchment or calf-skin, and the three divisions, when joined together, form what is called a drum or box sieve. The powder is made to pass through the meshes by gently agitating the sieve between the hands. A rough jarring motion will force through some of the coarser particles, and thus destroy the uniformity of the powder, and hence the common practice of tapping it frequently against the side of the mortar should be abandoned, unless the state of fineness is immaterial. Some substances, however, as magnesia, &c., which obstruct the pores of the cloth, must be

Fig. 46.



forced through in this manner, and even, if necessary, by a circular motion of the fingers over the interior surface of the cloth. This manipulation frees the meshes of the cloth from obstructions, but it must be carefully done, otherwise the safety of the cloth will be endangered. A sieve is also useful for the admixture of powders of uniform fineness.

6th. Levigation-is that mode of mechanical reduction which is practised by first rubbing the substance into a smooth paste, and then separating the finer from the coarser portions, by agitating the bruised matters with water. After a sufficient repose, the grosser and heavier portions subside, leaving the lighter particles still suspended in the water. This water, after decantation, gives a second deposit of an increased state of tenuity. The third or fourth decantation yields the powder of impalpable fineness. The time of repose between the decantations, unless great impalpability is required, should be limited, and only long enough to allow the deposition of the heavier portions. The coarse precipitates are collected together, second and as many more times as necessary, rubbed up as before, and treated with water, until all the lighter portions have been separated. This process applies only to substances unalterable by water. When uniformity of fineness is not all important, one washing even suffices, and can be accomplished in the mortar without the use of glasses. Alternate poundings and washings will eventually reduce and remove the whole contents of the mortar. In washing over gold and other metallic ores, where only the heavier portions are to be reserved, the water may be allowed to flow directly into the mortar, which being held in an inclined position, permits its exit, together with the fine dusty portions which are kept in suspension by trituration with the pestle.

This process of levigation is founded upon the different specific gravities of the coarse and fine bruised matters, and is, therefore, not only applicable for the separation of the particles of homogeneous matters, but also of equally fine matters of unequal densities. In the latter case it takes the name of *elutriation*.

All minerals for analysis, which have to undergo ignition with alkalies, should be previously levigated, in order that decomposition may be complete; for if the powder is not uniform the larger particles will escape decomposition.

Pulverization in this manner, by uniformly comminuting

the particles, promotes their equal expansion and the escape of contained moisture, and thus prevents the decrepitation of substances when heated.

The deposited powder must always be dried, by exposure, previous to subjecting it to any other process.

7th. Reduction by Granulation.-The reduction of metals to a pulverulent state is effected by fusing them in a crucible, and pouring the melted matter, from an elevation, in a thin stream, very gradually, into a bulk of cold water, which is, during the process, kept in constant agitation with a stirrer. The fineness of the resultant granules is proportional to the slowness with which the fused metal was poured into the water. It is more convenient to transfer the metal from the crucible into a ladle, and project it into the water from that more handy vessel, which enables a frequent change of the position of the descending stream, and thus prevents the formation of clots instead of smaller and more solid granules. The fusion of zinc for granulation must be in a covered crucible, otherwise it becomes oxidized whilst hot, and partially sublimes by exposure in an open vessel. Zinc may also be finely divided by being beaten, whilst hot, in a heated mortar.

The process of fusing metals and then agitating the melted matter in a wooden box until cool, reduces them to a state of minute division, but at the same time promotes their oxidation. For general purposes, however, it is not objectionable, and the particles of charred wood with which it becomes mixed can be separated by elutriation. The sides of the box are generally well chalked, to prevent any adherence of the metal;—this also is separable by elutriation.

# REDUCTION BY CHEMICAL MEANS.

8th. Division by Intermedia.—This mode is both mechanical and chemical, and applies particularly to the noble metals, in foil, which are difficult of pulverization. Honey, sugar, salts, &c., are the most usual media. By binding the particles together, it assists their minute division, and prevents their escape from the mortar. The addition of boiling water solves out the medium, without action upon the metallic powder, which then only requires to be thrown upon a filter and dried.

Phosphorus may be finely divided by fusing it, with alcohol,

# REDUCTION BY CHEMICAL MEANS.

over a water-bath, and shaking the contents of the flask until thoroughly cooled. The phosphorus subsides at the bottom in pulverulent form. Camphor, which is obstinate under the pestle, readily yields to its power when mixed with a few drops of alcohol or ether, to destroy its elasticity.

Silica may be precipitated from lime glass in a pulverulent form, by the digestion of that compound with hydrochloric acid. Silver is obtained in a powder by the decomposition of its nitric solution with a metallic copper rod; or of its chloride by metallic zinc. Proto-sulphate of iron throws down gold, in a finely divided state, from the solution of its muriate; and spongy platinum is formed by the dull ignition of the ammonia-muriate of that metal. These are instances of chemical division by purely chemical means. The extreme state of division thus obtained by the solution and precipitation of a solid body (and also by fusion, a chemico-mechanical process), cannot be effected by any purely mechanical power.

The sublimation of sulphur into flowers, as also of calomel into fine powder by means of large airy chambers, are instances of comminution by chemico-mechanical means;—the vaporized particles being prevented from reunion, at the moment of solidification, by the intervention of the cold air. So, likewise, in cases of division by hydro-sublimation, the intervention of aqueous vapor prevents the conjunction of the vaporized molecules. Dr. Joslin (*Silliman's Journal*, p. 48, vol. v.) treats of this subject in extenso.

# CHAPTER V.

### THE BALANCE.

A BALANCE may be considered the most indispensable implement of the laboratory, as affording the only means by which the chemist can accurately estimate the quantitative results of his investigations. The construction of this instrument for determining the relative weight (the measure of the force by which any body, or a given portion of it, gravitates towards the earth) of substances, is based upon certain mechanical principles, of which we proceed to give a brief explanation.

A balance consists of an upright shaft, supporting, by its immediate centre, an inflexible *lever* or beam, with arms of equal length and symmetry, to each of which is suspended a dish for the reception of the weights (the *power*), and the body to be weighed (the *resistance*). Of the three axes of the beam, that in the middle is the *fulcrum* or centre of motion, upon which it turns in a vertical plane. The other two axes are at the extremities of the arms. All three axes should be at right angles to the plane of motion, and parallel to each other.

The requisite conditions of a good balance.—One of the chief conditions of an accurate balance is a free suspension of the beam, in order that it may vibrate with the least possible friction. The two arms must also be precisely equal, so that when empty, or the weight in each dish is uniform, there will be a perfect equilibrium. The sensibility of a balance is proportional to the angle formed by the beam with the horizon, when a slightly greater weight is placed in one dish than in the other. This sensibility depends on the position of the centre of gravity of the beam with reference to the line of suspension; this centre must be below that line, but as near as possible to it, so that the slightest weight will cause the beam to oscillate freely.

As the inertia and friction are proportional to the weight of the beam, it must be made of material entirely free from imperfections, and so as to combine strength and inflexibility with lightness. It may be of solid steel, rolled brass, German silver, or of a malleable alloy of copper and tin, but not of cast metal of any kind. The upright support can be of brass, and the dishes and suspension frames of platinum.

The sensibility of the balance increases with the length of the arms, which should, however, have a certain limit, and be as nearly uniform as possible in every respect. When, through unskillful construction, the length of one arm is slightly greater than that of the other, in order to avoid the error in weighing which this defect would occasion, the body to be weighed is placed in one pan, and counter-balanced by weights in the other. The amount of weight required to restore the equilibrium after the withdrawal of the substance is its correct weight.

In order to avoid friction, the parts of contact should be

as few as possible, and the knife edges must be made of highly polished, hardened steel, and the beds or *planes* upon which they rest, of agate or flint. The accuracy of the balance will depend greatly upon the skill and precision with which these portions and the beam are elaborated.

A good balance, with 1 to 2000 grains upon each dish, should be sensitive to the one or two-thousandth of a grain.

"To obtain the greatest degree of uniform precision, it is requisite that the beam should be lifted from a state of rest, in a perfectly level position, and that the stirrups should be lifted, simultaneously, with their loads, from their rests or supports; also that the oscillations of the stirrups should be prevented or checked at the earliest moment; and, finally, that the whole system should be left at liberty with delicacy and exactitute, so as to remain in equilibrium, or vibrate as the case may be."

"To command the above conditions, the beam should be supported upon cones, at each extremity, adjusted level with each other, from which it is lifted, by a plane (and not a portion of a hollow cylinder, as is usual) which rises under its centre knife edge, and to which it is returned by its depression, the cones guiding the beam to the same position exactly from which it was elevated.

"The stirrups, in like manner, should hang upon hollow cones or V's, so as to be taken up from, and returned, invariably, to the same position.

"The beam should rest upon its cones, and the stirrups should be supported by their V's at such heights as to relieve entirely the knife-edges, with a sufficient space between them and their respective planes to permit inspection and wiping, when it may be needed. This construction admits of the placing of the weights, &c., and guards the knife-edges from the consequences of displacement during use.

"The beam should be raised by the elevation of the centre plane, subsequently lifting with it the stirrups with their weights and load, and all oscillation checked by platforms placed in the table under the centre of the stirrups, which should be made to rise simultaneously, and should be counterweighed to the requisite delicacy.

"The descent of these platforms, effected by the pressure of a finger on a lever conveniently placed, will leave the stirrups, &c., at liberty to vibrate, or bring the beam to a horizontal position, at the will of the operator; being a convenient, certain, and rapid method of manipulating, not equaled by any other arrangement, and, in fact, essential to a well-constructed balance."

These essential qualities of an accurate balance for the more delicate operations of the laboratory are comprised in that form of balance used in the United States Mint, and which "combines all the important advantages heretofore known with such improvements as have been the growth of their own experience." The possession of one of these instruments does away with the necessity of a separate balance, exclusively, for dry assays.

Fig. 47 gives a front view of this balance. We take our



description from the Journal of the Franklin Institute, vol. xiv.

Description.—A table, marked A, is furnished with leveling screws upon the front and back edge, and at each end, marked b. In Fig. 49, which exhibits different views of all the parts, the leveling screws are marked b, and their positions in the table (the view of the under side of which is given) are marked c.

The balance is intended to be placed upon a counter, or any other firm support, and the table leveled by means of the screws last described, its true position being indicated by a plumb-line and weight occupying the rear opening in the

# THE MINT BALANCE.

column (Fig. 49, C); the plumb-line and weight being marked d.

The column, marked C, Figs. 47, 49, contains the lifting apparatus, and supports the cap-plate, marked D. The capplate guides the lifting apparatus, and supports the V's, or hollow cones, for the stirrups, and is strengthened and stayed by braces, marked E; the section of which braces is cruciform, with circular ends, for firm bearing upon the plate and base of the column, to which they are secured by screws.

Figs. 48, 49, exhibit upper and under views of the table, column, plate, &c., also upper and lower end views of the column, showing the means of its attachment to the table and cap-plate.



The lifting apparatus consists of a winch-handle, marked f, Fig. 49, fitting upon a round shaft, g, with a feather, so as to admit of its convenient removal; upon this shaft is fitted a cam, h, also secured by a feather; the cam is carefully constructed, so as to give equal elevation to equal parts of its revolution; and upon the cam rests a roller, i, which turns upon a pin in the frame, j, intended to reduce friction, and give facility in raising the beam with its load.

The lifting frame, j, is forked cross-wise, so as to straddle the shaft and accommodate the cam and roller, at the same time that it allows the necessary vertical motion, without the possibility of being displaced; all of which is exhibited in the two views of the lifting frame marked j, which is also accompanied by sections in proximity to the parts which they are intended to explain.

The handle is so placed as to be on the left when the beam is down and at rest, and to the right when the beam is raised, in the act of weighing, and makes, together with the cam, more than three-fourths of a revolution, the cam having a very slight depression upon its upper, or highest point, into which the roller falls, maintaining it in its position when the beam is raised. It is then extended beyond the centre of the roller, so as to be stopped at the limit of motion, as exhibited h, Fig. 49.



The lifting frame is forked at the top for the accommodation of the beam. Upon it rests the plane, the top and side view of which are marked k, for the support of the centre knife-edge, secured to the frame by screws. In balances of ordinary construction, this plane may be made of hardened cast-steel; in finer instruments, of bronze, or brass, with an inserted block of polished agate, secured by fusible metal, or cement.

The position of the handle, lifting frame, &c., are exhibited with sufficient clearness in the front view, Fig. 47.

The cap-plate, views of the upper and under sides of which are given at D, Fig. 48, is constructed with horizontal spaces at the centre and each end. In the middle it is secured to the column by four screws, and to the braces B in the same manner, the holes for which are marked in all the views.

The square opening in the middle serves as a guide and

#### THE MINT BALANCE.

support to the lifting frame, which must be accurately fitted, so as to prevent any lateral play.

The horizontal spaces at the extremity of the cap-plate support short pillars terminated by cones, upon which the beam rests; these pillars are secured to the cap-plate by screws passing through it from the under side, the holes through which they pass being large enough to admit of the adjustment of the beam to its proper place, previous to their being permanently fastened down.

The details of these pillars are given at l, Fig. 49, the cones being constructed of cast steel, hardened and polished.

The same space also supports the V's, or guide supports of the hangers, different explanatory views of which are given in Fig. 49, the V's being marked m, and the hangers n. All these parts have been devised with reference to the simplest and most economical construction consistent with the requisite accuracy, and for affording the greatest facility in the final adjustment of the balance.

The most important part of the balance is the beam o; Fig. 49 exhibits side and top views. The projections marked q, are the supports of the beam when 'at rest; the conical cavities, indicated by dotted lines, being made to fit the cones marked l.

This form of beam affords facility in construction, being composed of straight surfaces, without ribs or curves; is well adapted to maintain its form when loaded; affords the least surface for accumulation of dust, and is readily wiped when it may be necessary. The means of adjustment for the length of arm is exhibited at r, Fig. 49.

It will be seen, that the needle of the balance, which is the subject of description, is pointed downwards, and there are good reasons for this disposition. In the first place it is directly before the eye of the operator, and, therefore, more convenient in use, than it is, when elevated above; again it may be made longer than the arms of the beam, and will, consequently, describe a larger arc, and thus give more distinct indications, whilst the whole arrangement need occupy no more space than is requisite for the other parts; and, finally, the needle is protected from external injury by the lifting-frame and column, in the centre of which it is placed.

The parts which remain to be described have been usually considered of minor importance, but experience has shown
that this estimate is scarcely a just one, inasmuch as they afford facilities for accuracy and rapidity, that leave no doubt of their value, and place them in a most important position in practice. The parts now alluded to, constitute the system by which the operator is enabled to find the equilibrium of which he is in search. It consists of the pedestals, as they have been termed, marked s, Figs. 47 and 48, and the parts connected with them, marked t, u, v and w, in Fig. 48; a light shaft, made of tubular iron, t, supported by pivots u, which pivots are screwed through a piece cast on the under side of the table, marked V; upon the ends of this shaft there are levers, W, upon the ends of which levers, when in place, the pedestals rest.

The remaining part of this system is a double armed lever, placed in the middle of the shaft, t, (represented in the engraving detached,) and marked x; it is connected by a pin, with the trigger, z, represented in its place in Fig. 48, with the same letter. Upon the other end of the lever, x, there is a weight, y, capable of adjustment by a screw upon which it traverses, so that it may be made to approach, or recede from the shaft, t.

The action of this system is easily understood; its whole object is to depress the platforms by sufficient force, applied by the finger, to the trigger, the counter weight returning them to their original position, after its removal.

It will be seen, by reference to Fig. 47, that the under sides of the stirrups have a space, represented by dotted lines, in which the platforms are placed, which allows the stirrups to oscillate within its limits, but beyond which they cannot move. This construction is intended to guard the hangers from displacement, and to prevent injury by too much movement of the stirrups, an accident very likely to occur, when the pans or weights are hastily removed, especially in the use of heavy weights or large masses.

The cavity, whose object was described in the last paragraph, forming the under side of the base of the stirrups, is turned as truly as possible in the form of a portion of a sphere, whose radius is its distance from the bearing of the knife-edge. The platforms are adjusted by means of the counter weight, so as to press lightly up against the stirrups, and to follow them when raised.

It is found convenient in practice to turn the handle of the

balance but a small portion of its movement, if the weights are not equal on opposite sides, a circumstance to be expected when searching for a weight. The heavy side will remain down, and the needle will indicate whether addition of weight, or its removal is requisite. These trials are continued until the platforms follow up the whole lift, the needle remaining opposite the middle line of its scale, until the handle is stopped by its limit of motion, where it remains. The finger, then, by pushing down the trigger, will depress the platforms, when smaller weights are employed until the needle indicates equilibrium.

In this balance there is little or no embarrassment from oscillation, because the stirrups immediately accommodate themselves to the position of the weights, the light pressure permitting them to take any position required by the load; nevertheless, having sufficient power, from their pressure, to prevent any swinging. If from any cause the stirrups should be in motion, three consecutive depressions of the platforms, will bring them to a state of rest, with absolute certainty, and with a loss of time so short as to be entirely immaterial.

The stirrups are connected with the hangers, by a rod, which is double-jointed, as near to the hangers as possible, so as to allow perfect freedom of motion; at the same time, so well fitted as to allow no change of position in the parts. On the lower ends of these rods, there are screws and nuts, to regulate the height of the stirrups, together with a jam nut, to prevent any change after the adjustment has been satisfactorily made.

The bases of the stirrups are designedly made small, requiring the use of a dish on the one side, and a platform for weights on the other. This dish and platform being made of equal weight, renders the use of a counter weight unnecessary, and as the balance cannot be used without both, the liability to mistakes from this cause is entirely avoided.

Kater's and Robinson's balance, which, though more complicated, and less preferable for other reasons to the preceding, is the most popular balance for estimating minute quantities with precision; and, indeed, for all the weighing operations of delicate research. When carefully preserved, it retains its sensibility for many years. Fig. 50 represents one made by Mr. J. P. Duffey, of Philadelphia, who has acquired great skill and accuracy in the construction of fine balances. An

## KATER'S AND ROBINSON'S BALANCE.

Fig. 50.



improvement which he has added to those of recent manufacture, is both simple and useful. It consists of an elastic spring, A A, Fig. 51, serv-

ing as a support for the dishes when the balance is at rest; and at the same time so arranged, that by the depression of the thumb lever suitably attached, the dishes are thrown off their



supports, and the beam put into action simultaneously. We are indebted for our description to Lardner's Elements of Mechanics.

"The beam of this balance is only ten inches long. It is a frame of bell-metal in the form of a rhombus. The fulcrum is an equilateral triangular prism of steel, one inch in length; but the edge on which the beam vibrates is formed to an angle of  $120^{\circ}$ , in order to prevent any injury from the weight with which it may be loaded. The chief peculiarity in this balance consists in the knife-edge, which forms the fulcrum, bearing upon an agate plane throughout its whole length; whereas in the other balances the whole weight is supported by portions only of the knife-edge, amounting together to one-tenth of an inch. The supports for the scales are knife-edges, each six-tenths of an inch long. These are each furnished with two pressing screws, by means of which they may be made parallel to the central knife-edge.

"Each end of the beam is sprung obliquely upwards and towards the middle, so as to form a spring through which a pushing screw passes, which serves to vary the distance of the point of suspension from the fulcrum, and, at the same time, by its oblique action, to raise or depress it, so as to furnish a means of bringing the points of suspension and the fulcrum into a right line.

"A piece of wire, four inches long, on which a screw is cut, proceeds from the middle of the beam downwards. This is pointed to serve as an index, and a small brass ball moves on the screw, by changing the situation of which the place of the centre of gravity may be varied at pleasure.

"The fulcrum, as before remarked, rests upon an agate plane throughout its whole length, and the scale-pans are attached to planes of agate, which rest upon the knife-edges, forming the points of support. This method of supporting the scale-pans, we have reason to believe, is due to Mr. Cavendish. Upon the lower half of the pillar, to which the agate plane is fixed, a tube slides up and down by means of a lever which passes to the outside of the case. From the top of this tube, arms proceed obliquely towards the ends of the balance, serving to support a horizontal piece, carrying at each extremity two sets of Ys, one a little above the other. The upper Y's are destined to receive the agate planes to which the scale-pans are attached, and thus to relieve the knife-edges from their pressure; the lower to receive the knife-edges themselves, which form the points of suspension of the pans, consequently these latter Ys, when in action, sustain the whole beam.

"When the lever is freed from a notch in which it is lodged, a spring is allowed to act upon the tube we have mentioned, and to elevate it. The upper Ys first meet the agate planes carrying the scale-pans, and free them from the knife-edges. The lower Ys then come into action and raise the whole beam, elevating the central knife-edge above the agate plane. This is the usual state of the balance when not in use: when it is to be brought into action, the reverse of what we have described takes place. On pressing down the lever, the central knife-edge first meets the agate plane, and afterwards the two agate planes, carrying the scale-pans, are deposited upon their supporting knife-edges.

"A balance of this construction was employed by Captain Kater, in adjusting the national standard pound. With a pound troy in each scale, the addition of one-hundreth of a grain caused the index to vary one division, equal to onetenth of an inch; and Mr. Robinson adjusts these balances so that with one thousand grains in each scale, the index varies perceptibly on the addition of one-thousandth of a grain, or of one-millionth part of the weight to be determined."

A balance of this or the preceding kind, necessarily costly (\$85 to \$100) from the great care required in its construction, is only needed for the weighing of minute quantities of matter in scientific researches, and where it is desirable to estimate the least appreciable differences of weight. An instrument well calculated for all the ordinary purposes of analysis, is the Berlin balance, Fig. 52.

Those manufactured by E. N. Kent, New York, are guaranteed, when loaded with 50 grammes in each pan, to turn with  $\cdot 005$  grammes, or  $\tau \overline{\tau} \cdot \overline{\tau} \cdot \overline{\sigma} \overline{\sigma} \overline{\sigma} \overline{\sigma}$  th part of the weight. Its cost, with an extra pan for taking the specific gravity of bodies, and glass case, containing a drawer with divisions to receive the different parts of the balance, and thus render it portable, is thirty dollars.



In many processes, and, indeed, in some few instances of analysis, for example of gold ores and of vegetable matters, one or more constituents of which are only obtainable, even in minute quantities, from large amounts of the material, it is necessary to have a second balance calculated to weigh from a quarter of an ounce to five pounds with such precision that one or two grains will turn the dish when loaded with its greatest weight. Fig. 53 represents a balance of this kind, made after a Tralles beam, by Duffey, of this city. It consists of two brass dishes, A A, suspended by loops, D D, which rest upon the steel knife-edges at the extremities of the beam C. The beam is supported by the knife-edge in its centre as at E, and the whole balance is suspended to an upright, crooked at its top, by the hanger B. Annexed to the centre of the beam is a long vertical needle, which, following the vibrations of the beam, serves to indicate the least oscillation, which is rendered more perceptible by an ivory seg-



ment situated behind its point, and divided into degrees. This balance is placed in the room D, Pl. 2, upon the table adjoining that upon which is the finer balance. The support to which it is suspended may be either of wood or metal. To prevent damage to the knife-edges, the dishes, when the scales are not in use, should be unhung, and the whole balance kept covered with a linen cover, distended over a wire frame work, which may be suspended by a cord upon a pulley, and counterpoised so as to admit of being readily raised or lowered.

Preservation of the Balances.--Each of the finer balances should have a separate table, and these tables a permanent position in a close, well-lighted room, expressly appropriated for the purpose. The top of the table must be of hard wood and perfectly horizontal; and to secure the table itself against the slightest jarring motion, it may be tightly fastened to the floor by iron clamps and screws fitted to its legs. Great

## PRESERVATION OF THE BALANCE.

care is requisite to have it plumb. Of the two drawers which it should contain, one is for the spatulas, spoons, crucible tongs, papers, watch glasses, and other implements used in weighing; the other may be fitted deskwise, and furnished with slips of paper, or a porcelain slate and pencil, to afford convenience in recording the weights. A polished cast-iron slab about six inches square, upon which to set the heated crucibles and promote their cooling by conducting off the excess of heat previous to weighing, may be considered a necessary accompaniment to the table. In order to preserve its brightness, it should be encased in a woolen bag and kept within the drawer when not in use; or it may be enclosed in a frame with a sliding cover, and fastened to the top of the table near to one of its corners. The cover which protects its surface from oxidation can readily be drawn out whenever it is required to use the slab.

It is not sufficient for the preservation of the delicacy of the balances, that the room in which they are kept should be dry and tight. Vapors, aqueous and corrosive, will in divers ways find entrance into the apartment, and so, therefore, besides the precaution of lacquering the brass and steel parts of the balance, the instrument should be enclosed in a sufficiently. capacious mahogany or walnut case, with sash doors in the front and back, and sash windows at either side. The doors should be always kept closed and fastened by their buttons when the balance is not in use, else the entrance of dust and moisture will impair its accuracy. An additional and very effectual precaution against oxidation by moisture, is to keep constantly within the case a capsule containing some absorbent matter, as fused chloride of calcium or carbonate of By an occasional renewal of the absorbent matter, potassa. the atmosphere within the case can be kept very dry. The multiplication of door-ways is to afford facility for the passage and weighing of long tubes, which have to be placed across the pan. The divisions in the drawer at the bottom of the case must be lined with velvet, and so arranged as to receive the different parts of the balance, and allow its transportation without the least risk of damage.

In order to preserve the edges of the knives, the beam should always be thrown out of action when the balance is not in use, otherwise their constant contact with the planes and the pressure of the balances, as well as the sudden addition of a heavy weight, will injure their delicacy. Duffy's support, with its thumb lever outside, before mentioned, affords a means of not only preventing these contingencies, but also of communicating motion to the beam, without the necessity of opening the doors, and consequent exposure of the balance.

The three or four feet upon which the case rests have screws passing through them. These serve to give the balance a perfectly horizontal position, even upon an uneven surface—the level being obtained by raising or depressing either of the screws, as the case requires, and as will be indicated by the two spirit levels which should be fitted to the pedestal of each balance.

The position of the balance should be with due regard to light, but while placed near the window, to afford facility in perceiving the slightest oscillations of the needle, it should be free from any sudden actions of the solar rays, which, by producing unequal expansion of the different parts would occasion inexact results in weighing.

The balances should be cleansed and adjusted whenever they have become inaccurate, for it is almost impossible for even the best balance to retain its sensitiveness indefinitely. This work should rather be confided to a manufacturer of balances, as it requires both skill and experience. Slight discrepancies in the weight of the two dishes can be temporarily compensated for by adding to that dish which is deficient, sufficient weight to restore its equilibrium.

## CHAPTER VI.

#### THE WEIGHTS.

THERE are three sets of weights requisite, of which, one for the common scales of the operating room should be avoirdupois, and range from eight or more pounds downward to an ounce or less. These weights are for the rougher operations of weighing, and may be of cast iron, with a coating of black japan to protect them against oxidation.

#### THE BALANCE-THE WEIGHTS.

For the second balance (Fig. 52) the weights must be of brass, in the form of short cylinders f, Fig. 56, with knobs at the top, and should range from 25,000 to 10 grains, decreasing by fives, in the series to 500, as follows: 25,000, 20,000, 15,000, 10,000, 5000, 1000, 500; and from that point in ratio as follows: 400, 300, 200, 100, 50, 30, 20, 10, so as to make altogether 15 weights; the whole to be enclosed in a suitable box with a hinged cover.

These latter weights, though required to be accurately adjusted, are not necessarily so nicely precise as those for the analytic balances (Figs. 47, 50). They should always be subdivided after the decimal system, so that ten of the smaller weights will make one of the next highest class. This arrangement (so that they perfectly agree with each other of the same denomination) will render it unimportant whether they be grain or French gramme weights, the two kinds almost exclusively used in scientific research.

If they are grain weights, the series should range from 5000 grains to  $\frac{1}{1000}$ th of a grain, as follows:—5000, 3000, 2000, 1000, 500, 300, 200, 100, 50, 30, 200, 100, 50, 30, 20, 10, 5, 3, 2, 1, .1, .2, .3, .5, .05, .03, .02, .01, .005, .003, .002. The gramme weights range from one centigramme to one milligramme. The divisions of the gramme (the standard unit of the French weights) are the decigramme= $\frac{1}{10}$ th gramme; the centigramme is the centigramme =  $\frac{1}{100}$ th gramme; the milligramme =  $\frac{1}{1000}$ th gramme are the decagramme = 10; the hectogramme = 100; the kilogramme = 1000; and the myriagramme = 10,000 grammes. The table below shows the relative value and proportions of the French decimal and troy and avoirdupois weights.

Metrical or Decimal Weights.	
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		Equiv. in Troy	Equi	v. m avoirdupois
Names.	Equiv. in grammes.	grains.	77 .	weight.
			103.	02. grs
Milligramme	.001	.0154		
Centigramme	.01	.1543		20, 20
Decigramme	.1	1.5434		· · · · · · · · / ·
Gramme	1.	15.434	, 7	1, 10 , 9 ,
Decagramme	10.	154.3402	200	04 45
Hectogramme	100,	1543.4023	1, 3	34. 12.153
Kilogramme, or	Kilo 1000.	15434.0204	° 12	34 12:173
Myriagramme	10000.	154340.2344	22	03 .12.
		E = 2 2	7 7	

All of the weights for the fine balance must be adjusted

with the nicest accuracy, and before being used should be compared with others of attested correctness. Of the French weights those from the milligramme to the gramme should be of platinum; and so, also, of the grain weights, those from the ten grain weight downwards should be of the same metal. Palladium being of but half the specific gravity of platinum, and similar to it in other respects, is sometimes used for the fractional grain weights, because of the greater relative surface which it presents. The remaining larger weights, to save expense, may be of brass, and, to preserve them from oxidation, should either be covered with a thin coat of lacquer or else galvanized with gold or platinum.

Each set should contain duplicates of the 10, 2 and 1 grain weights; triplicates of the .2, .1, .02, .01, and quadruplicates of the .001 grain weights; in many instances, especially in taking specific gravities, these additional weights are indispensable.

In order to preserve the weights free from dust and oxidation, they must be encased in a close box with hinged cover and fastenings. The interior of the box is divided into compartments, lined with velvet to prevent abrasion of the surfaces of the weights, each of which should have a separate division. The edges of every compartment should be marked in ink with the value of the weight it contains; and the weights themselves must have their denominations stamped upon them. The series must be so accurately adjusted, that the difference between any one of the large weights and the combined number of smaller ones, equal to it, shall not be perceptible in a balance turning with one-tenth of a milligramme.

To test their accuracy, take any two of them of the same denomination, convey them, with the fork or forceps, to the balance, and place one in each dish. If the beam upon being put in action is in perfect equilibrium, the weights are uniform, and can serve as standards. Now, for further verification, place both together in one dish, and in the opposite pan add enough of smaller weights to equal that of the two combined. If the beam still retains its equilibrium, when put in action, the weights may be considered correct.

As frequent handling of the weights with the fingers would tarnish them, and otherwise injure their value, a small fork and a pair of forceps are necessary accompaniments to each set of weights. The fork, represented by Fig. 54, made of either ivory, horn or wood, and being intended for raising

### THE BALANCE-THE WEIGHTS.

the brass weights, has its notches at either end of different size, so as equally to accommodate the knobs of

both the larger and smaller weights. A small pair of elastic forceps, Fig. 55, of brass or plated, or polished steel, with platinum, or ivory points, serve for the smaller platinum weights, which, for convenience of handling, should be turned up at one corner, as at g, Fig. 56.

The box should be closed after each weighing, and, to preserve it from the corrosive vapor that may be floating about,



should be kept in the drawer of the balance table.

Fig. 56, represents a box of weights and all the necessary



appurtenances. The ribs  $a \ a \ a$ , fitted to the interior of the top, by pressing against them when the box is closed, keep the weights in their places. The fork and forceps are shown in place at  $b \ b$ . The channel d, kept always covered with a thick glass plate, contains the platinum weights, an extra quantity of the smaller of which are kept in the cavity e. These cases and the weights, accurately adjusted and finely finished, can be purchased of either Kent or Duffey.

Small weights, to supply the place of such as may be accidentally lost, can be made by first determining the weight of a given length of wire of uniform thickness throughout, and then dividing it into perfectly equal parts;—the number of the divisions indicates the fraction of the original weight which they represent as a whole; for instance, if the wire weighed ten grains; and is divided into 10 or 20 portions, each fraction will represent a grain or half grain, accordingly. By using wire of greater thickness, weights of augmented value can, in like manner, be made and adjusted.

Shot, of which there should be a box of the several sizes kept in the balance table-drawer, are convenient counterpoises for tubes, capsules, crucibles, watch glasses, and other receptacles for substances to be weighed.

# CHAPTER VII.

### WEIGHING.

THERE are certain preliminaries to be observed in all delicate weighing operations, of which the most important is to ascertain whether the balance is in order, as regards equilibrium and freedom of oscillation. To do this, each dish should be loaded nearly to the full extent of the power of the balance. If, when the beam is put in action, there is no perceptible variation in the dishes, the equilibrium is perfect. For further verification, there should be an exchange of loads, from one dish to the other, and the beam again set in motion. The recovery of the equilibrium, after the cessation of the vibrations indicates the correctness of the balance. The need of more than a milligramme for the analytic balance, or of  $\frac{1}{10}$  th of a milligramme in the more delicate balance, to restore a deficiency in either dish, should condemn the instruments for quantitative examinations, unless previously adjusted. This is done by the addition of bits of tin-foil to that dish which is lightest. When, however, the balance is carefully used, and by but one operator, it will be only necessary to reassure himself of its equilibrium in those weighings where absolute accuracy is all important. Be careful, however, in adjusting, as well as in weighing, that the weights in the pan do not overload the balance and make it set, an effect the more prompt, in proportion to the greater accuracy and sensibility of the balance. The setting, which makes one scale appear heavier than the other, is a permanent depression of the lowest pan by the slightest impulse to the exactly horizontal beam of a

surcharged balance. Hence the necessity of loading the balance within the limit of its maximum power.

In all weighing operations, the counterpoising of substances is more speedily attained, and with less injury to the balance, by systematically following a weight, which is removed from the pan as too heavy, by the next in succession, until equilibrium is obtained. Thus, in balancing a watch glass, if the 50 grain weight drags the beam, replace it by the 15; if this is still too much, use the 10 weight; and if this is too little, make up the deficiency with the smaller weights, added consecutively, and decreasing gradually their denomination as the counterpoise is approached.

To preserve the accuracy of the balance, it should be put out of action upon every addition, removal, or substitution of weights. As a precaution against error, the weights must always be removed from the pan and spread upon white paper to be counted; and to verify the aggregate, in putting them away, their denominations should also be compared with their value, as marked upon the vacancies which they occupy in the box in which they are kept. The slips of paper and porcelain slate, in the table-drawer, serve to make notes of their amount, which should be done before placing them in the box.

A provision against inaccuracy, from very slight inequality of the arms of the beam or imperfect equilibrium from other causes, is the invariable use of the same pan for the reception of the substance to be weighed. By this practice, notwithstanding the difference in the weights of the two dishes, the ratio being kept uniform, the quantities will be proportionably augmented or decreased, so that the products of analyses can be as accurately estimated as in a perfect balance. An alternate use of the pans for the weights and the substance to be weighed, will, on the contrary, lead to results too high or low, as the case may be. We, however, obtain, in this way, only the *relative* weight of the substance, and not its *absolute* weight, which requires a perfect balance.

Borda proposes to avoid the errors of inaccurate balances, by first taking the tare of the substance with that or any other counterpoise, and afterwards substituting, in its stead, weights sufficient to restore the equilibrium, which was disturbed by its withdrawal from the dish. The sum of these added weights represent exactly that of the substance being weighed. This mode is termed *double weighing*, and affords very nice results even in balances with disproportioned beams.

A modification of the above method is, supposing, for instance, that five grains of a substance are required, to place twenty grains' weight in one dish, and a small capsule in the other, and then to establish equilibrium by adding, to the latter, the requisite number of very fine shot. This done, remove 15 grains from the first dish, and introduce into the capsule sufficient of the substance, to be weighed, to compensate for their loss.

Weighing of Solids .- The balance being in perfect order and repose, the next step is to counterpoise the vessel in which the substance, which should in no instance be placed upon the naked dish, is to be weighed. Circular disks of highly glazed paper are sometimes used as recipients, but being attractive of moisture, are preferably replaced by a watch glass or crucible of platinum or of porcelain. The recipient being placed upon the pan, appropriated exclusively for the purpose, is then accurately counterbalanced by shot or fragments of metal. In analyses, the counterpoise must be preserved for future references. They may be either wrapped in paper or enclosed in paper pill-boxes, but in either case must be labelled. In delicate analyses, to avoid error, the tare of the vessel should be estimated in weights, and their amount immediately noted down, to be afterwards subtracted from the combined weight of it, and the substance weighed. The tare of the drying tubes, or of Liebig's and other apparatus used in organic analysis requiring to be weighed, to prevent mistakes, should be labelled upon the implements themselves with which it corresponds. This done, the substance is introduced into the recipient, if in lumps, by means of a pair of forceps with platinum points; if in powder, with an ivory, horn, or platinum spoon or spatula, accordingly as it may be inert or corrosive. The blade of the spatula should never be of steel, as it is so liable to oxidation. A slip of very thick sheet platinum, one inch in width and two inches long, fastened in a wooden or metallic handle, is generally used. Its usefulness for this purpose may be increased by alloying it with a very minute portion of silver, which increases its elasticity in an eminent degree. The weights are added to the opposite dish, and always after throwing the beam out of action, through the side door of the case, which must be im-

#### WEIGHING OF CORROSIVE SUBSTANCES.

mediately closed after each addition. If, when the balance is lifted from its supports, by depressing the thumb lever extending without the case, the index needle turns rapidly towards the dish opposite to the weights, the balance must be put in repose, another weight added, and the motion of the needle again examined. This operation should be repeated upon the addition of each weight, however small. As the pans approach equilibrium, the vibrations of the needle decrease in rapidity, and a little experience and observation will enable one to hit the right point after one or two trials. When any given quantity of a substance is to be weighed, the requisite weight should be placed in the dish first, and the substance if in powder deposited in the other dish with a spoon or spatula, until an accurate counterpoise is obtained, taking care however, to bring the balance at rest upon each addition of material, which may be made to fall in very minute quantities from the spatula by gently tapping against its handle with the finger.

Those substances which are hygrometric, should be weighed in a covered vessel; for instance, between two watch glasses, or in a small tube with a ground stopper, which may be held in an upright position by a twine loop slipped over the suspending wire of the pan, or by a cork and wire stand, as shown by Fig. 57.

The more delicate balances have for this purpose, for that of organic analysis and of weighing substances in water, a supplementary pan, with a hook beneath, for convenience of suspension. This pan, which descends from the beam only one-half the distance of the other, is shown by Fig. 62.

After the weighing is completed, the weights, as before directed, are withdrawn with forceps, placed upon a piece of white paper, and their several amounts added together; the total gives the weight of the substance in the opposite dish.

Covered vessels are also requisite for those corrosive substances the exhalations from which would be injurious to the balance, and impair its accuracy.

Substances should never be weighed whilst hot, even in closed vessels, otherwise the ascending current of air thus produced, together with an unequal expansion of one arm of the beam, will give inaccurate results. A crucible, therefore, which has been over the lamp for the ignition of its contents,

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### WEIGHING OF LIQUIDS.

should be first cooled by standing upon the iron slab accompanying the balance table, otherwise its *hot* weight, not corresponding with its *cold* weight, would, in estimating the result, lead to an error in the real weight.

Weighing of Liquids.—The nature of liquids, especially their temperature and volatility, have an important influence upon the precision of the results.

Non-volatile liquids may be weighed in a counterpoised capsule, watch glass, flask or tube. Those vessels which have spherical bottoms are supported in the pans upon cork rings, which are readily made by hollowing out a cork and bevelling its upper edges interiorly. If the recipient is tall, it requires

Fig. 57.

a stand to maintain it in an upright position. This stand, shown by Fig. 57, is nothing more than a disk of cork b, with the wire catch a, fastened to it. An excellent substitute, is a broad cork, with a hole in its centre, corresponding with the size of the tube, and bored smoothly and nearly through the cork with the cork-borer hereafter to be described. The use of the twine loop, before mentioned, is less safe and convenient for

suspending these vessels.

The liquids are conveyed to the recipients in dropping tubes or pipettes. This mode allows their gradual addition, and in small quantities. The pipette, for small quantities, is

Fig. 58.

drawn out at its lower end, as shown by Fig. 58. The tapering end of this tube, being placed in the liquid, as soon as the latter has risen, interiorly to its external level, place the thumb upon the upper orifice of the pipette, withdraw it from the liquid, and convey it to the counterbalanced recipient in the scale dish. Holding it immediately over this recipient, you then remove the finger and allow the liquid to be driven out by atmospheric pressure, either in

nothing more than a tube of a quarter inch diameter,

a thin stream or drops, according as the capillary orifice of the thin end of the pipette is larger or smaller. Remember that, as pressure of the surrounding fluid is the cause of the liquid's ascension into the tube, its ingress is proportional to the depth to which the pipette enters the containing vessel.

When the pipette contains more liquid than the required

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#### WEIGHING OF LIQUIDS:---PIPETTES.

quantity, the flow can be arrested as soon as the given weight is counterbalanced, by quickly replacing the finger upon the upper orifice of the tube. After removing it from over the pan, the surplus can be emptied into the original vessel.

Any excess of the liquid, preventing a perfect adjustment, may be removed from the recipient with an empty pipette in the same manner as it was introduced. With a little precaution in the management of the pipette, the flow may be made so gradual that it can be arrested as soon as the vessel has received the required quantity. The insertion of slips of bibulous paper serves to withdraw any slight excess, unless the liquid is corrosive and acts upon paper, in which case, a glass rod must be substituted. The insertion of this rod into the liquid, and its subsequent withdrawal, causes a loss thereto of the small adherent quantity, and thus we have a means of accurately weighing any required quantity of liquid.

If the quantity of liquid to be weighed is large, the form of the pipette must be as shown by Fig. 59. The bulb in its centre serves as a reservoir for the required charge. If the nature of the liquid does not render the operation disagreeable to the manipulator, the pipette may be quickly filled by suction with the mouth. This plan, however, is objectionable, as being liable to introduce moisture. A much better method is to cover tightly the larger or upper end of the pipette with a caoutchouc bottle. By compressing this bottle with the hand, a partial expulsion of air ensues, and the liquid, into which the pipette is plunged, rushes in quickly to fill the vacuum. The bottle, being again distended by the upward pressure of the interior atmosphere, prevents the exit of any drop of liquid, until it is forced out by compressing the bag a second time.

Some chemists prefer pipettes of syringe construction, Fig. 60. Like the afore-mentioned, they must be invariably of glass. The liquid is drawn from its original container by immersing the taper end therein, and raising the piston;-the liquid mounts into the cylindric reservoir until filled. The depression of the piston, by the pressure of the

Fig. 59.

finger upon the top of the handle, causes the exit of the liquid with rapidity proportional to the power applied.

The stock of pipettes should consist of several sizes. After being used, they should be carefully laid across a porcelain plate, there to remain until the completion of the weighing, when they must be well rinsed out previous to being returned to their places in the drawers.

All volatile substances must be weighed in small, closely stoppered flasks, tubes or other vessels, previously counterpoised. To insure accurate results, care must be taken that the temperature does not favor volatilization. Fuming liquids should, if possible, be measured.

To preserve the balance, as much as possible, from their corrosive action, the recipient should be removed from the pan upon each addition or withdrawal of any portion of its charge, and tightly closed again before being returned. The slender tube, Fig. 99, for small quantities, and the syringe for larger proportions, proper implements for conveying the liquids to the

are the proper implements for conveying the liquids to the balance.

Very deliquescent substances, such as are not checked in their liquefying tendency by the greatest practicable dryness of the balance case, and are not alterable by water, should be weighed in solution. First, pour in a sufficiency of water in the counterpoised recipient and note its weight. The increase of weight given to this water by the addition of the deliquescent body, is the actual weight of the latter, and the solution can be used in the analysis instead of the solid. In some instances, according to the nature of the substance, alcohol may be substituted for water.

Weighing of Gases.—In weighing gases, it is necessary, in order to obtain nice results, to guard against the least variations of temperature, pressure, and humidity of the atmosphere. Gases are readily estimated by means of a very sensitive balance, though the old plan, by measurement of volume in graduated vessels, is accurate and easily executed.

Gases are weighed in counterpoised balloons, which must be thoroughly cleaned and dried, and wiped exteriorly and interiorly, so as to remove every particle of grease, dirt or dust. The balloon is then to be connected by a coupling cock fitted

Fig. 60.

to its neck, with an air-pump (Fig. 32) or syringe, and exhausted of its air. To insure the expulsion of all remnants of gas from a former weighing, the balloon should be subjected to repeated alternate exhaustions and airings. The exhausted balloon is then to be attached to a graduated bell-glass, also fitted with a coupling cock, as shown by Fig. 61. This bellglass is the reservoir of the gas which is received or

collected over (see PNEUMATIC TROUGH) water or mercury; the latter fluid, giving more exact results, is used for those gases which are soluble in the former. Communication between the balloon and bell-glass, being made by opening their connecting cocks, the gas rushes from the latter into the former by force of atmospheric pressure upon the mercury. When the balloon is filled, the flow is to be stopped by closing the cocks. A delay of several minutes is necessary (see MEASUREMENT AND TRANSFER OF GASES) to allow the temperature within the bulb to become that of the external air, that the level within and without the bell-glass may be equalized, and the quantity of gas noted. The balloon must then be detached, and again weighed with care and accu-

racy. The difference between its present and original weight, is that of the volume of gas which it contains.

In the weighing of gases, it is indispensable to note the temperature and barometric pressure, and to observe all other conditions and precautions requisite in the MEASUREMENT OF GASES.

Those gases which are without action upon mercury, ought to be collected over that metal, for most gases, by contact with water, absorb more or less of its vapor, according to the temperature, and, therefore, to insure correct results, it is generally preferable to purify the gas of moisture *previous* to weighing it. This is done by passing it through a tube (see DESICCATION OF GASES) containing fused chloride of calcium, or some other absorbent substance.

It must be recollected, however, in the desiccation of gases, by transit through tubes containing absorbent matter, that the quantity of dry gas, entering into the globe, is less than that received from the bell-glass, the volume of vapor abstracted in its passage. To determine the amount of this loss, "observe the temperature of the moist gas, and correct its

Fig. 61.

### WEIGHING OF GASES.

volume by the pressure of thirty inches of mercury. Then, by the table below, ascertain the proportion of vapor which was present in the volume which left the jar, and subtract it from the corrected volume;—the remainder will be the volume of dry gas which has entered the globe."

The table, taken from Faraday, "exhibits the proportion by volume of aqueous vapor existing in any gas standing over or in contact with water, at the corresponding temperatures, and at mean barometric pressure of thirty inches."

40°		.00933	51°	.01380	61°		.01923	710		.02653
41	_	.00973	52 -	.01426	62		.01980	72		.02740
42		.01013	53	.01480	63		.02000	73		.02830
43		.01053	54 -	.01533	64		.02120	74		.02923
44		.01093	55 -	.01586	65	_	.02190	75		.03020
45	_	.01133	56 -	.01640	66		.02260	76		.03120
46		.01173	57	.01693	67		.02330	77		.03220
47		.01213	58 -	.01753	68		.02406	78	_	.03323
48		.01253	59	.01810	69		.02483	79		.03423
49		.01293	60 —	.01866	70		.02566	80	-	.03533
50		.01333								

This table is also useful for determining that part of the volume and weight of a moist gas, due to aqueous vapor after it has been weighed without previous desiccation, for as it "includes any temperature at which gases are likely to be weighed, the proportions in bulk of vapor present, and consequently of the dry gas, may easily be ascertained. For this purpose the observed temperature of the gas should be looked for, and opposite to it will be found the proportion in bulk of aqueous vapor at a pressure of 30 inches. The volume to which this amounts should be ascertained and corrected to mean temperature. Then the whole volume is to be corrected to mean temperature and pressure and the corrected volume of vapor subtracted from it. This will leave the corrected volume of dry gas. It has been ascertained, in a manner approaching to perfect accuracy, that a cubic inch of permanent aqueous vapor corrected to the temperature of 60°, and a mean pressure of 30 inches, weighs 0.1929 grains. The weight, therefore, of the known volume of aqueous vapor is now easily ascertained, and this being subtracted from the weight of the moist gas, will give the weight of the dry gas, the volume of which is also known.

"As an illustration, suppose a gas standing over water had been thus weighed, and that 220 cubic inches at the

temperature of 50° Fahr., and barometric pressure of 29.4 inches had entered into the globe and caused an increase in weight of 101.69 grains. By reference to the table it will be found that at the temperature of 50°, the proportion of aqueous vapor in gas standing over water is .01333, which in the 220 cubic inches will amount to 2.933 cubic inches, which corrected to the temperature of 60°, becomes 2.942 cubic inches. The whole volume corrected to mean temperature and pressure will be found to equal 219.929 cubic inches, from which, if the 2.942 cubic inches of aqueous vapor present be subtracted, it will leave 216.987 cubic inches as the volume of dry gas at mean temperature and pressure: 2.942 cubic inches of aqueous vapor weigh .5675 grains, for 2.942  $\times 0.1929 = 0.5675$ ; this subtracted from 101.69, the whole weight, leaves 101.1225 grains, which is the weight of the 216.987 cubic inches of dry gas; and by the simple rule of proportion, therefore, it will be found that 100 cubic inches of such gas, when dried and at mean temperature and pressure, will weigh 46.603 grains.

"It is not necessary in this experiment that the globe or flask be perfectly exhausted of air before the gas be admitted, all that is necessary in that respect being, that the quantity of gas which enters, and the corresponding increase of weight, be known. For the same reason it is not necessary that the globe be filled, provided the quantity which does enter is ascertained upon the graduation of the jar when the level is the same inside and outside; and that no alteration of the quantity in the globe be allowed before the weighing is completed. The state and quantity of the gas are estimated in the jar, and it is there that the temperature and pressure should be attended to. It is essentially necessary that the temperature of the globe over the water should have been steady for some time before the experiment be made, and that it do not change until the gas has entered the globe and the stop-cock is securely closed. After that, a little variation of temperature is of no consequence, so that nothing passes into or out of the globe until the conclusion of the experiment. The globe, as before said, should be clean and drv."

# CHAPTER VIII.

## THE DETERMINATION OF SPECIFIC GRAVITY.

BODIES which are of uniform bulk may vary in density, and thus give rise to a difference in their specific gravity—the relation of their weight to their volume. The density of bodies is estimated by certain standards; that for solids being pure distilled or rain water (=1.000) at 60° F. The number, therefore, expressing the specific gravity of a body is the number of times it is heavier or lighter than an equal volume of water. For example, if two bodies of equal bulk differ in density in the ratio of one to two, the latter is said to have twice the specific gravity of the former, and so in proportion. Therefore, "the volumes being equal, the densities of bodies are directly as their weights; or, the weights being equal, the densities are inversely as their volumes."

"Thus, if a cubic centimetre of iron weighs 7.8, while an equal volume of water weighs only one gramme, 7.8 is the specific gravity of iron." Hence, to find the density or specific gravity of a solid substance, its absolute weight must be divided by the weight of an equal volume of water.

SPECIFIC GRAVITY OF SOLIDS. By means of the Hydrostatic Balance.—The two principal operations for estimating the specific gravity of a solid heavier than water, are: First, to weigh it accurately in air; and secondly, to weigh it in water. The weight at each weighing must be immediately noted in the record book.

All of the finer balances are provided with a supplementary pan (Fig. 62), for these weighings in fluids. Though necessarily but one-third the depth of the regular pans, it is accurately made so as to exactly counterbalance either of them, and when adjusted to the beam, in its stead, to maintain perfect equilibrium. The hook beneath the pan is a convenience for the suspension of the body to be weighed. This arrangement permits the weighing of the body in air, and subsequently in water, without disturbing it or the balance. The process is as follows: Suspend the body to be weighed by a very fine platinum wire or unspun silken thread, to the hook at the bottom of the supplementary pan, and adjust this dish to that side of the beam from which the regular dish has been removed to give place to it. There are other substitutes for platinum and silk, but being more permeable and less capable of furnishing a very fine and strong thread, they do not afford such nice results in delicate experiments. This done, take the weight of the body in air, observing the requisite precision in WEIGHING, and note it down in the record book without delay. To take the weight in water, it is now only necessary to convey a beaker glass containing that liquid, immediately under the dish, and carefully to immerse therein the suspended body. This vessel must be of diameter sufficient to allow a free play of the body without contact with its sides. Fig. 62, represents the whole arrangement.

In introducing the body into water, particularly if it presents rough surfaces, the air attaches itself in bubbles, which must be removed with either a camel's hair pencil or wooden stick. These precautions being duly observed, put the balance in action and take the weight of the body, and immediately record it. The apparent loss of weight represents the weight of the bulk of water which the body displaces, and hence we have the requisite data upon which to calculate its specific gravity, viz., its weight in air and the weight of its own bulk of water.



Thus, for example, the body weighs :---

In air . In water	e :	•	•	373 grains. 341 "	
	Loss			32 "	

By following the rule, and dividing the total weight by the loss of weight in water, thus  $373 \div 32$ , we have 1.165 as its specific gravity or density.

If the body is lighter than water, a weight of known magnitude and density is joined with it to sink it. The weight may be a capsule, and form a part of the furniture of the balance for this especial purpose. It should be cullendered to allow the free escape of the globules of air adherent to the

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body, after receiving which, it should be suspended to the short pan as before directed.

In this, as in the previous instance, the weight required to re-establish the equilibrium disturbed by the immersion of the body in water, expresses the weight of the volume of water displaced.

"The rule, therefore, is—from the difference between the weight of the two in water and their weight in air, subtract the difference between the weight of the heavy solid in air and its weight in water; the remainder is the weight of a quantity of water equal in bulk to the light solid, from which the specific gravity of the substance may be obtained by simple proportion.

"As an example, suppose the following case:-

- 1. The weight of the light solid in air . . 12 grs.
- 2. The weight of the heavy solid in air . 22 "
- 3. The weight of the heavy solid in water . 19 "
- 4. The weight of both tied together in water 8 "

"Then, from the weight of both in air (12+22) 34 grs. Deduct the weight of both in water . . . 8 "

"And from the remainder deduct 22 - 19 = 3 "

"Which gives the weight of the bulk of 23 "

water displaced by the light body alone  $\int$  .

"The following proportion then affords the specific gravity of the body:----

as 23:12::1.:0.5217."

## (Parnell.)

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If the substance is porous or in powder, its specific gravity is better estimated by weighing it in the bottle (Fig. 64), after the precaution of disengaging all adherent globules of air; otherwise it must, in following the preceding process, be weighed in a capsule, counterpoised first in air and afterwards in water. The water also should, before being used for this purpose, be freed of any contained air by pouring it several times from one vessel to another.

When the body is soluble in water, it must be replaced by some other liquid of determined specific gravity, which is without action. Olive oil, spirits of turpentine and alcohol, are applicable, one or the other, for most cases. "The specific gravity of the substance is then found by the following proportion,—As the density of water is to the density of the liquid used, so is the density of the substance in relation to the liquid in which it is weighed as unity, to its density compared with water as unity."

"By the above described process, we find how much a certain quantity of fluid weighs which has the same volume with the body to be weighed, and when once the specific gravity of the fluid is known, it is necessary to ascertain the weight of an equal volume of water."

"Let it be assumed, that a piece of salt, which is insoluble in oil of turpentine, weighs 0.352 gram., and displaces when put into the glass 0.13 gram. of oil of turpentine. The specific gravity of this fluid is 0.8725; an equal volume of water will therefore weigh—"

 $\frac{0.13}{0.8725} = 0.149$ , and the specific gravity of the salt is, there-

fore,  $\frac{0.352}{0.149} = 2.36.$ 

The preceding mode of taking the specific gravity of substances is founded upon the Archimedean law of hydrostatics, "that the weight of a substance in any medium is less than its absolute weight, by the weight of the bulk of the medium which it displaces, obviously its own bulk." It is, however, objectionable, as being liable to give inaccurate results, and, therefore, we proceed to speak of a better method.

2d. By means of a Stoppered Flask.—In this mode of weighing, which is very available for taking the density of minute bulks of matter, and applies to bodies either heavier or lighter than water, the same attention to temperature is requisite, as in the preceding process.

The glass bottle in which the body is to be weighed, is of form, as shown by Fig. 64. Its stopper should be round, slightly conical, and accurately ground. To

facilitate the egress of any excess of water, in case of expansion by heat, and to enable it to sink to a uniform depth in all positions, the centre of the stopple should be perforated. The precision with which the bottle and its stopper are manufactured, has an important influence in bringing out

Fig. 63. Fig. 64.



an exact result. This bottle is especially adapted for taking the specific gravity of liquids, and its capacity may be from 100 to 1000 grains of distilled water.

Three weighings are required in taking the specific gravity by this mode. The flask is first filled with water, so as to exclude all air, then conveyed to the balance and accurately counterpoised. The body to be examined is then placed in the same pan with the flask, and the balance being again set in action, the weight of the body is expressed by the additional weight necessary to produce equilibrium; and that of the body and flask by the whole weight. The substance being examined, is then placed in the flask, which is again weighed, after having been wiped clean, to free it from the displaced fluid which has flowed over its sides. The third weighing is to determine the quantity of water, which is a volume equal to its own bulk, displaced by the immersed body. Having thus obtained the requisite data, we calculate as follows:

"The glass vessel with water weighs . . . 13.52 grms. The body . . . . . . . . . . 4.056 "

. 17.576

"

Both together .

"If, after throwing the body into the bottle, putting the stopper in, and weighing the whole together, we find it to be 17.316 grammes, the weight of the water forced out by the body must be 17.576 - 17.316 = 0.26 gram.; consequently the specific gravity of the body is  $\frac{4.056}{0.26} = 15.6$ ."

3d. By means of the Areometer.— The areometer is an instrument which may be conveniently substituted for the balance in taking the specific gravity of solids. That known as Nicholson's is most generally used. Muller describes the apparatus and the mode of manipulating with it, clearly and concisely, as follows:

"To a hollow glass or metal body v, Fig. 65, a small heavy mass l (a glass or metal sphere filled with lead) is suspended, and superiorly there is attached to it a fine stem supporting a plate c, on which small bodies and weights may be laid. The instrument floats vertically in the water, because its centre of gravity is very low in consequence of the weight l. The instrument is so arranged that the upper part of the body v projects above the water. If now we lay the body whose specific gravity we would ascertain upon the plate

c, the instrument will descend, and by adding additional weight, we may easily make it sink to the point f, marked generally by a line on the rod. We remove the mineral or other substance we have been using, and substitute in its place as many weights as will again make the instrument sink to f. If, in the place of the mineral, we have had to lay on n grains, the weight of the mineral is equal to n grains.

"If, in this manner, we have ascertained the absolute weight of the mineral, the n grains must be again removed, and the body laid in a basket placed between v and l. The instrument would now again sink to f if the body laid in the basket



had not lost weight by being immersed in the water: we must, therefore, lay on the plate the weight m grains, that the body may be immersed to the mark. In this manner we obtain the absolute weight of the body n, and the weight of an equal volume of water m; the specific gravity we seek is, therefore n

therefore,  $\frac{n}{m}$ .

"If, for instance, we have to determine the specific gravity of a diamond, we must lay it on the plate and add sufficient weight to make the whole sink to f. If we find after removing the diamond, that we must lay on 1.2 grains to cause the areometer to sink again to the same point, the absolute weight of the stone would be 1.2 grains. These weights must be again taken away and the diamond laid in the basket; then, in order to make the instrument sink to f, we must add 0.34 grains more; the weight of a volume of water equal in volume to the diamond is, therefore, 0.34 grains, and the specific gravity required is  $\frac{1.2}{0.34} = 3.53$ ."

SPECIFIC GRAVITY OF FLUIDS.—There are three modes of determining the specific gravity of fluids, taking precedence in the order in which we name them; by the stoppered flask, the hydrometer and gravimeter. The first method yields the greatest accuracy, and is that used in all nice investigations. By the Flask.—Any small ground stoppered flask or vial will answer for the purpose. It must first be brought to the temperature of 60° F., then accurately weighed, and after the removal of the stopper, filled with distilled water of corresponding temperature. The stopper is then to be inserted, and the water that it displaces wiped from the sides of the vessel, which when dry is again carefully weighed. Its increase of weight expresses that of the bulk of water which it contains, and to save time and trouble, should be marked with a diamond upon the neck of the flask to serve for future experiments.

Glass flasks of this description are made by the manufacturers especially for this purpose. Their capacities are arranged so as to exactly receive a given quantity of distilled water expressible by weight in round numbers. The sizes vary from 100 to 1000 grs., but in each instance they have a diamond scratch on their necks showing the measure of their contained weight of water. They have been previously described at p. 115, and are represented by Figs. 63, 64. The smaller one is most used because of greater facility in handling. Moreover the quantities of fluid under examination are generally limited, and hence the convenience of a small flask both on this account, and because it is more easily weighed in a delicate balance.

For the more volatile liquids, the perforated stopple should be replaced by a solid one, otherwise loss by evaporation may occasion incorrect results.

If the chemist prefers purchasing a flask to graduating one himself, it must be verified as above before being used, and if the weight of its contents of water does not correspond with the mark on the neck of the flask, or of the flask and water combined with that of the weight generally accompanying it as a convenient counterpoise, then the flask is not to be relied on, and should either be corrected or rejected.

In either case the flask must be thoroughly cleansed, and after each experiment should be repeatedly rinsed with distilled water, so that it may be perfectly *clean and dry* when wanted for the next operation. In emergencies, the interior may be dried by placing the flask upon the sand-bath; in this case, however, it will be necessary to allow its temperature to fall again to  $60^{\circ}$  before using it.

Distilled water at 60° F. is the standard by which to estimate the specific gravity of liquids. To take the density of

## 

a liquid, equal bulks of it and water are taken at the balance. The flask having been graduated, its weight and that of the bulk of its contents of water are already known; it is, therefore, only necessary to fill it with the liquid under examination at 60° F., to the mark upon its neck, and after inserting the stopper, carefully weigh it. Divide the weight thus found by the weight of the water (as indicated on the flask) and you obtain the specific gravity of the liquid. For example:— The clean and dry flask weighs 400 grains.

do do filled with pure water, at 60° 900 "

Deduct the first from the latter and you obtain the weight of the water = 500. Supposing, then, that the same bulk of the liquid weighs 412 grains, then  $412 \div 500 = 0.824$  its specific gravity.

If the capacity of the flask is 1000 grains of water, and one of that size, Fig. 63, may well be used, when the stock of the liquid under examination is not limited, the process is still easier. It is then only necessary to fill it with the fluid and weigh it. The weight obtained expresses its specific gravity; thus, taking mercury for example, a bulk of that metal equal to the bulk of 1000 grains of water, is 13,500 grains, and, therefore, these latter numbers express its density, taking care to advance the decimal point three figures to the left, if the water is taken as 1 instead of 1.000.

The vessel must, previous to weighing, be invariably wiped dry exteriorly with a linen cloth, and to avoid any communication of heat from the hands, they should be gloved.

For determining the density of very minute quantities of rare liquids, it will be necessary to have the aforementioned bottles of miniature dimension, or else to replace them by glass bulbs.

If the fluid is volatile and readily vaporizable, it should, in being raised to the proper temperature, be heated over a spirit lamp, in a test tube; taking care to keep the finger over its mouth, during the heating and cooling, so as to prevent its being unclosed.

By the Hydrometer.—Hydrometers do not give very accurate results, but they are convenient when time is an object, and no great precision is requisite. Their action is based upon the hydrostatic law, "that a floating body displaces its own weight of the liquid in which it swims." The instrument consists of a glass stem A, with an air bulb, B, beneath, properly ballasted with mercury or shot, D.

Fig. 66. Fig. 67.

The depth to which the hydrometer will sink in a liquid is proportional to its rarity, for the denser the liquid, the less of it will be displaced. A properly graduated scale inserted within the stem or spindle, allows the appreciation of the density of a liquid by the greater or less depth to which it sinks therein. The form of this instrument is shown by Figs. 66, 67. They are constructed (Morfit's Applied Chemistry) with different scales accordingly, as they are intended for liquids rarer or denser than water. The scales for those which are rare run from zero (at the bottom of the stem) upwards. The graduation of the scale for liquids denser than water is reversed.

Areometers are variously graduated for different liquids, thus-

Those	for	ether :	mark	upwards	from	10 to	$50^{\circ}$
"	66	spirits	66	- 66	66	10 to	80
66	66	salts	66	downwar	ds "	0 to	40
66	66	acids	66	66	66	0 to	75
66	66	syrup	s ""	66	66	0 to	36

The following table shows the specific gravity numbers corresponding with Baumè's areometric degrees :----

## HYDROMETERS-MANNER OF USING.

Liquids denser than water.						Less dense	than w	vater.	
Degrees.	Specific gravity.	Degrees.	Specific gravity.	Degrees.	Specific gravity.	Degrees.	Specific gravity.	Degrees.	Specific gravity.
0 1 2 3 4 5	$\begin{array}{c} 1.0000\\ 1.0066\\ 1.0133\\ 1.0201\\ 1.0270\\ 1.0340 \end{array}$	26 27 28 29 30 31	$\begin{array}{c} 1.2063 \\ 1.2160 \\ 1.2258 \\ 1.2358 \\ 1.2459 \\ 1.2562 \end{array}$	52 53 54 55 56 57	$\begin{array}{c} 1.5200\\ 1.5353\\ 1.5510\\ 1.5671\\ 1.5833\\ 1.6000 \end{array}$	10 11 12 13 14 15	$\begin{array}{c} 1.0000\\ 0.9932\\ 0.9865\\ 0.9799\\ 0.9733\\ 0.9669\end{array}$	36 37 38 39 40 41	$\begin{array}{c} 0.8488\\ 0.8439\\ 0.8391\\ 0.8343\\ 0.8295\\ 0.8249\end{array}$
6 7 8 9 10	$1.0411 \\ 1.0483 \\ 1.0556 \\ 1.0630 \\ 1.0704$	32 33 34 35 36	$\begin{array}{c} 1.2667 \\ 1.2773 \\ 1.2881 \\ 1.2992 \\ 1.3103 \end{array}$	58 59 60 61 62	$\begin{array}{r} 1.6170 \\ 1.6344 \\ 1.6522 \\ 1.6705 \\ 1.6889 \end{array}$	16 17 18 19 20	0.9605 0.9542 0.9480 0.9420 0.9359	42 43 44 45 46	0.8202 0.8156 0.8111 0.8066 0.8022
11 12 13 14 15	$\begin{array}{c} 1.0780 \\ 1.0857 \\ 1.0935 \\ 1.1014 \\ 1.1095 \end{array}$	37 38 39 40 41	$\begin{array}{r} 1.3217\\ 1.3333\\ 1.3451\\ 1.3571\\ 1.3694 \end{array}$	63 64 65 66 67	1.7079 1.7273 1.7471 1.7674 1.7882	21 22 23 24 25	0.9300 0.9241 0.9183 0.9125 0.9068	47 48 49 50 51	0.7978 0.7935 0.7892 0.7840 0.7807
16 17 18 19 20	$1.1176 \\ 1.1259 \\ 1.1343 \\ 1.1428 \\ 1.1515$	42 43 44 45 46	$\begin{array}{r} 1.3818 \\ 1.3945 \\ 1.4074 \\ 1.4206 \\ 1.4339 \end{array}$	68 69 70 71 72	1.8095 1.8313 1.8537 1.8765 1.9000	26 27 28 29 30	0.9012 0.8957 0.8902 0.8848 0.8795	52 53 54 55 56	0.7766 0.7725 0.7684 0.7643 0.7604
21 22 23 24 25	1.1603 1.1692 1.1783 1.1875 1.1968	47 48 49 50 51	$1.4476 \\ 1.4615 \\ 1.4758 \\ 1.4902 \\ 1.4951$	73 74 75 76	1.9241 1.9487 1.9740 2.0000	31 32 33 34 35	0.8742 0.8690 0.8639 0.8588 0.8538	57 58 59 60 61	0.7656 0.7526 0.7487 0.7449 0.7411

The hydrometer is used with a tall glass jar (Fig. 68), which serves as a recipient for the liquid to be tested. After having perfectly cleansed it of grease and dirt with a cloth, it is to be placed in the jar and the liquor, first brought to  $60^{\circ}$  F., added. When it becomes stationary, note the degree at which it stands. For verification, raise it an inch or more out of the liquid and then let it gradually sink back again. If it reaches the same point as before, the first observation was correct. In reading the divisions on the scale, do not take that line where the liquid rises in wetting the stem of the instrument, but note it at the real level, which is the curvature

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Fig. 68.

produced by the ascending motion of the liquid against the sides of the spindle.

The hydrometers graduated by Baumè's process are generally used. Those made by F. A. Greiner & Co., Berlin, are the most reliable.

For information, in detail, upon the construction of the different kinds of hydrometers, see Encyclopædia of Chemistry, and M'Culloch's "Investigations relative to Cane Sugar and Report on Hydrometers."

By Nicholson's Gravimeter. — "The specific gravity of liquids may also be determined by Nicholson's areometer, Fig. 65. As the instrument always sinks so far that its weight, added to the weight upon the plate, is equal to the mass of liquid displaced, we may, by the aid of this instrument, ascertain how much a definite volume of water weighs. It is necessary, however, to know the weight of the instrument itself. Suppose this weight to be n, we must lay on some additional weight to make the instrument sink to f; if we designate this addition by a, then is n + a the weight of water displaced.

"If we immerse the instrument in another liquid, we must lay on another weight b in the place of a, to make the whole sink to f; b will be greater than a if the liquid be denser, and less than a if it be lighter than water. The weight of the liquid displaced is n + b; but its volume is exactly as great as the volume of the mass of water, whose weight is n + a, because the areometer has sunk equally deep in both cases.

"Suppose the instrument weigh 70 grains, we must add 20 grains to make it sink in water, and 1.37, that it may sink to the point f in spirits of wine; then the specific gravity of spirits of wine is  $\frac{70 + 1.37}{70 + 20} = 0.793$ ."

SPECIFIC GRAVITY OF GASES.—The extreme lightness of gases and vapors renders it inconvenient to compare their weight with that of an equal bulk of water, and consequently air is taken as the standard.

The mode of taking these specific gravities is thus concisely and clearly described by Parnell, "From the careful experiments of Dr. Prout, it appears that 100 cubic inches of atmospheric air deprived of carbonic acid and aqueous vapor weighs 31.0117 grains, at 30 inches of the barometer,

### SPECIFIC GRAVITY OF GASES.

and at the temperature of  $60^{\circ}$  F.; from which observation it is easy to calculate the absolute weight of any bulk of a gas from its specific gravity. Thus the specific gravity of chlorine is found to be 2.47; to find how much 100 cubic inches of that gas weigh at mean temperature and pressure, we make use of the proportion,

as 1.: 2.47 :: 31.01 : 76.59;

therefore 100 cubic inches of chlorine weigh 76.59 grains.

"The simplest method of obtaining the specific gravity of a gas is the following:-The object is to ascertain the weight of a bulk of gas equal to the bulk of a known weight of air. For this purpose, a light glass globe, furnished with a stopcock, is very accurately weighed, when full of air; then exhausted of its air, by connecting it with an air-pump, and weighed in the vacuous state. The weight of the air withdrawn by the exhaustion is thus ascertained. The globe, still vacuous, is connected with a jar containing the gas which is to be weighed, at the water or mercurial trough; the jar having a stop-cock at its top, into which the stop-cock of the globe can be screwed air-tight. On gently opening both stop-cocks, a quantity of gas rushes from the jar into the exhausted globe, equal in bulk to the air withdrawn by the exhaustion, if the surface of the liquid within the jar be brought to the level of that without in the trough, and the temperature of the air and the barometric pressure have not varied during the experiment. The stop-cock being closed, the globe is detached from the jar, and weighed. The difference between its weight when containing the gas, and when vacuous, is the weight of a bulk of the gas equal to the bulk of air whose place it occupies, the weight of which has already been determined.

"Suppose the globe to lose 10.33 grains by exhaustion of air, and, when exhausted, to gain 15.78 grains by admitting carbonic acid gas; then, assuming 1. as the density of air, we have the proportion,

as 10.33 : 15.78 : : 1. : 1.527;

the specific gravity of carbonic acid gas is, therefore, 1.527.

"Although thus simple in principle, the operation in its details is one of extreme delicacy. From the facility with which gases undergo a change in their bulk through variations of temperature and pressure, it is obvious that if the temperature and barometric pressure vary during the course of the experiment, corrections must be made. As an illustration of the necessary corrections, suppose the bulk of air to weigh 12 grains at the temperature of  $60^{\circ}$  F., and under a pressure of 30 inches bar.; and the same bulk of the gas whose density is required to weigh 20 grains, but at the temperature of  $50^{\circ}$ F., and under a pressure of 28 inches bar. The points to be determined here are two:—

"1. Considering the volume of the air withdrawn and the gas admitted as 1., at the observed temperatures and pressures, what would be the volume of the gas at the temperature and pressure at which the air was weighed?

"And, 2, having obtained that volume, what is the corresponding increase or reduction in the *weight* of the gas?

- "(a) A volume of gas equal to 1. at  $50^{\circ}$  F. is equal to 1.019 at  $60^{\circ}$  F.
- "(b) A volume of gas equal to 1.019 at 28 inches of the barometer is equal to 0.951 at thirty inches.

"A volume of the gas, therefore, equal to 0.951 weighs 20 grains; a volume of air equal to 1. at the same temperature and pressure weighing 12 grains. Then, if 0.951 vol. weighs 20 grains, 1 vol. should weigh 21.03 grains: and

as 12:1::21.03:1.75;

1.75 is, therefore, the density required.

\* "1. For Changes in Bulk by Pressure.—The volume which a gas should possess at one pressure may be calculated from its known volume at another pressure, by the use of the following proportion —As the pressure to which the gas is to be corrected is to the observed pressure, so is the observed volume to the volume required. In the example in the text (b), the pressure to which the gas is to be reduced is 30 inches, the observed pressure 28 inches, and the volume is 1.019. Then, as 30:28:1.019:0.951.

"2. For Changes in Bulk by Temperature.—From the very recent experiments of M. Regnault, it appears that a volume of gas expands by heat  $\frac{1}{4b}$  of its bulk for each degree Fahrenheit. Hence, the volume of a gas at 0° F. being 1, at any higher temperature it is found by the formula  $1 + \frac{\text{Temp. Fah.}}{459}$ . The determination of the volume of a gas at one temperature from its known volume at another temperature may be attained by the following formula:—Let t be the temperature Fahrenheit at which the volume of the gas is observed; t' the temperature Fahrenheit to which the volume of the gas is to be reduced; x the observed volume at t; and x' the volume at t' required;

Then 
$$x' = \frac{(459+t') \times x}{459+t}$$
.

"3. It is frequently necessary to combine corrections both for temperature and pressure. In such a case, as in the example in the text, the reduction of volume is first made for temperature, and that corrected volume is afterwards reduced according to the pressure. "The state of dryness of a gas is another circumstance which interferes with its volume; for which reason, due care should be taken to insure either the perfect dryness of the gas, or its complete saturation with moisture. In the latter case, the temperature must be noticed, and the observed volume reduced according to the proportion of aqueous vapor capable of existing in the gas at the observed temperature. The proportions of vapor by volume contained in 1 vol. of the saturated gas for temperatures between  $40^{\circ}$  and  $80^{\circ}$  F. are expressed in the table at *page* 110. A cubic inch of aqueous vapor corrected to the temperature of  $60^{\circ}$ , and at a pressure of 30 inches, weighs 0.1929 grains.

"The preceding method of obtaining the density of a gas still requires a slight correction from another circumstance, when the temperature and pressure differ considerably at the time of weighing the air and at the time of weighing the gas; but one so trifling that it may, in general, be neglected. The necessity of this correction arises from the impossibility of obtaining a perfect vacuum in the globe; and the remaining small quantity of air may occupy a different space when weighed with the gas, to that which it occupied when the globe was weighed with air; and consequently, the bulk of the gas admitted into the globe is not the same as the bulk of the air withdrawn. If the amount of rarefaction of the air in the exhausted flask is observed, by means of a barometer gauge attached to the air-pump, the amount of the remaining air may be calculated when the weight of the quantity withdrawn is ascertained; then the alteration to which it would be subject in bulk by changes of temperature and pressure may also be estimated, and a due allowance made on the bulk of the gas admitted into the globe."

When the gas is corrosive in its action, as in the case of chlorine, the balloon with its metallic cock must be replaced by a glass flask with a nicely fitting ground stopper. This flask is to be adjusted to a drying tube connected with the vessel in which the chlorine is generated. The bent end of the drying tube entering the flask should reach to its bottom. The disengaged gas in passing through the tube parts with its moisture, and reaching the flask descends to the bottom, and displaces the air, which is expelled through the interstices at the mouth around the tube. When the chlorine itself begins to escape, it is evidence that all the air has been displaced, and the flask is then to be slowly and gently detached from the apparatus and hermetically closed with its ground stopper.

SPECIFIC GRAVITY OF VAPORS.—There are two modes of determining the specific gravity of vapors, the one devised by Gay Lussac (*Pelouze and Fremy's Chimie Générale*, vol. ii., *Traité de Manipulations Chimiques, par A. Bobière*, vol. ii. p. 467), and the other the preferable one of Dumas. It is as follows:

Take a glass globe of about 12 to 16 oz. capacity, with a long slender neck, wash it with distilled water, and carefully dry it, either by slight warmth or by means of the exhausting syringe and chlorcalcium tube, Fig. 69. After the balloon is



perfectly dry, its neck is to be drawn out to a narrow tube 6 or 8 inches long, and bent nearly at a right angle, as shown at a, Fig. 70. The tip is then to be removed with a file, and the mouth of the tube rounded (not closed) over the blow-pipe flame. The globe full of air is now weighed, with great precision, and afterwards warmed to expel a portion of its air. This done, its beak is immediately dipped into the liquid or melted\* solid matter, and as the air within contracts by the cooling of the bulb, which may be hastened by dropping ether on its exterior, the fluid is drawn up. When the requisite

\* If the solid body is not fusible, a given weight of it is introduced into the globe, previously dried. The neck is then drawn out, the end removed and placed in the balance. By deducting its weight from that of the whole balloon, you obtain the weight of the balloon full of air.

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#### SPECIFIC GRAVITY OF VAPORS.

quantity, say 100 to 150 grains, has entered, the globe is at once enclosed in a wire basket b, Fig. 70, and introduced into



a water, saline, metallic or other bath, the temperature of which exceeds the boiling point of the liquid,  $50^{\circ}$  or  $60^{\circ}$ . The wooden support *e*, to an arm of which is suspended the thermometer *c*, keeps the globe firmly fixed in the bath.

The bath is brought to ebullition, and as soon as it rises above the boiling point of the substance, a jet of vapor escapes through the tube, and as soon as it ceases, the point is sealed up over the blow-pipe flame, observing at the same time the temperature of the bath and the barometric pressure.

The globe thus closed is then withdrawn from the bath, washed, dried, and again weighed.

To determine the capacity of the balloon, its tube is dipped into mercury, and its point broken under the surface of the metal, which immediately rushes in and fills the vacuum caused by the condensation of the vapor, and should occupy the whole interior. It is evident that the volume of mercury represents the volume of the vapor at the noted temperature, and this volume is determined by transferring the mercury to a graduated tube, and marking the number of cubic inches or centimetres which it occupies.

We thus have all the data necessary for calculating the specific gravity of the vapor, having determined, experimentally,—

### MEASURES AND MEASURING.

- "1. The weight of the globe and air at ordinary temperature and pressure;
- "2. The weight of the globe and vapor filling it at the temperature of the bath, and under ordinary pressure; and,
- "3. The capacity of the globe.
- "Having these results, we obtain by calculation,-
  - "1. The weight of the empty globe (by knowing the capacity of the globe, the weight of the air filling it can be calculated, which, deducted from the weight of the globe and air, leaves the weight of the globe when vacuous);
  - "2. The weight of vapor filling the globe at the temperature of the bath (by deducting the weight of the empty globe from the weight of the globe and vapor); and,
  - "3. The weight of air filling the globe at the temperature of the bath, and at the pressure at which the globe was sealed with the vapor.

"The last calculation is made according to rules given in the note, page 124; having performed which, the density of the vapor required is obtained by the simple proportion,—As the weight of air filling the globe at the temperature of the bath is to the weight of vapor filling the globe at the same temperature, so is 1 to the density required."

# CHAPTER IX.

### MEASURES AND MEASURING.

Measuring of Fluids.—When great accuracy is required in the estimation of fluids, their weight is determined; but in ordinary operations, the amount of their volumes is obtained by the employment of vessels purposely prepared and graduated with care and precision.

These vessels or *graduates* as they are called, are generally of two forms, those for the larger operations being cylindrical, as shown by Fig. 71. This shape combines both strength and convenience. For the smaller (ounce or drachm)

### MEASURING OF FLUIDS.

graduates, the conical form, Fig. 72, is preferable, as giving greater facility by its smaller surfaces, for accurately estimating minute volumes.



Graduation.—For the large measures, the imperial pint is the usual integer. To graduate a vessel to this extent, take a glass balloon, Fig. 73, counterbalance it and weigh therein accurately one pint imperial (8750 grs.) of distilled water, at the temperature of 62° F., and at 30 inches of barometric pressure. After the vessel has remained undisturbed upon a level shelf, sufficiently long for its contents to acquire a smooth steady surface, scratch upon the neck the exact level to which the liquid rises. The narrower the neck of the flask the greater the facility in noting this point without liability of error. This weighed quantity of water is then to be transferred to the proof glass, under process, either of such a form as shown in Fig. 71, or in Fig. 74, the latter, however, being shorter and wider, is preferable for large sized graduates. After the water has settled, and presents a smooth calm surface, scratch its level accurately upon the exterior of the glass, either with a diamond point or a sharp file. Thus you obtain a pint measure, to graduate which into its subdivisions of ounces and drachms, it is only necessary to take the pro rata weights of the fractions of the pint, and proceed in manner as above directed. So likewise, the vessel can be graduated to pint divisions, in number as many as its capacity will admit, by multiplying the weights of water, and adding them to those previously measured, noting the level of each with the diamond.

The imperial pint is larger than the wine pint of 16 fluid-

ounces, in the ratio of 6 to 5, and, therefore, its subdivisions must number 20. This makes a discrepancy, the inconvenience of which can be remedied by having a second scale upon the same glass, showing their relative values. The only disadvantage is the trouble of a second graduation, which is, however, compensated for in the convenience of the first scale, each division of which, unlike the fluidounce of the wine pint, represents a fluidounce exactly, weighing one ounce avoirdupois of distilled water.

The plan of graduating the pint, itself estimated as above, into its subdivisions by apportioning its height into the requisite number of equal parts, by means of a rule, will only answer for vessels of uniform diameter throughout, and which are only intended for the grosser operations of measuring.

Those graduates, which are intended for nice purposes, should also have a third scale graduated in cubic inches. The cubic inch equals 252.458 grains of distilled water, at temperature and pressure the same as above. As there is sufficient room upon the glass for all of these scales without the necessity of crowding them together—there should be an equal interval between them.

To graduate a vessel to the litre of the French standard, substitute 1 kilogramme for the 8750 grains distilled water, and proceed as above, making the subdivisions pro rata.

The graduates and cubic inch bottles are less to be relied on when purchased than when carefully graduated by the operator himself, and they should never be used in important experiments without having been previously verified.

For the graduation of the ounce and drachm measures, and, indeed, all vessels of small diameters and capacities such as tubes and the like, the divisions of which must necessarily for want of space closely approximate to each other, mercury is much preferable to water. Mercury gives a more level and distinct surface than water, and not being attracted by the sides of the vessel, allows a greater accuracy in making the subdivisions, especially in very narrow tubes. The addition of one grain of lead to every 4000 grains of quicksilver improves the mercury for this purpose, but it must be otherwise pure and free from dross and film. A cubic inch of pure mercury, according to Faraday, weighs 3425.35 grains, at  $62^{\circ}$  F.

There should be a series of these graduated glasses, ranging from a double pint down to a drachm.

For the tubes and other vessels used in analytic research, the decimal divisions are both convenient and necessary. If a cubic inch is to be divided into tenths and hundredths, the former are graduated by the space occupied in the tube by the one-tenths (342.50 grs.) of a cubical inch of mercury, and each tenth division coincident with the level of the metal within, is marked upon the scale. So also, in like manner, are the hundredths graduated by substituting 34.25 grs. (the hundredth of a cubic inch at  $62^{\circ}$ ) for the 342.50 grs. mercury.

To give a clear idea of the mode of preparing a measure with mercury, let us suppose that a tube is to be graduated to cubic centimetres (of the French standard). In the first place a narrow strip of white paper, with a line ruled down its centre, is to be pasted lengthwise upon the side of the glass to be graduated, the length of the paper of course corresponding with the height of the glass. 13.59 grammes of mercury are next to be accurately weighed out, and this quantity, which represents a cubic centimetre, is to be poured into the tube, held vertically by a support similar to A, in Fig. 79. After the vessel has stood long enough for the liquid to become quiet and assume a smooth surface, its level is noted down, and its corresponding height marked with ink upon the paper slip. The space which this bulk of quicksilver occupies in the tube equals a cubic centimetre, and when accurately noted, may serve as a standard for the graduation of vessels of larger capacity; for these cubic centimetral divisions can be multiplied, merely by multiplying this given bulk of mercury, and noting and marking upon the paper, the level of each addition as its surface becomes smooth. Ten times the above weight of mercury gives a decimetral division, and one-tenth of it a millimetral division, and thus we have an easy mode of enlarging or diminishing the subdivisions of the scale.

By having the tubes accurately graduated so that their divisions exactly correspond with the weights of the balance, we acquire the convenience of calculating at once the weight of gases from their measured volume.

The plan of consecutive weighings, involves a good deal of trouble and labor where large vessels are being prepared, and hence, in such cases, the convenience of this mode of multiplying the divisions by an accurately adjusted measure.

In marking the scale, let those lines designating the tenths

#### GRADUATED VESSELS.

extend in width a little beyond those denoting the twentieths, and these latter, in their turn, a little beyond those expressing

Fig. 75.

70

- 60

- 50

40

-30

.20

10

the hundredths. Fig. 75 represents a graduated glass with a properly written scale, upon which the tenths are shown by figures.

As these glasses are to be standard graduates for a variety of purposes in the laboratory, the scale should be indelible, or etched upon the glass. For this purpose the paper scale must be covered with a thin transparent film of melted white wax. When the wax has cooled and hardened, the lines and figures are graved out of the paper with a sharp pointed style or buren, and the exposed surfaces of the glass subjected to the action of fluohydric acid, as directed at p. 68. This done, and the wax scraped off, the etched portions show out distinctly, and are better defined than if they had been scratched, as is sometimes done, with the

diamond point or file.

Be careful that the subdivisions conform accurately among themselves, and in the aggregate precisely with their integer. The volumes as expressed by the lines on the scale should also exactly agree with their corresponding weights, for upon these conditions depends the accuracy of results.

Tubes for eudiometry, Fig. 76, and proof glasses for alka-



limetry, Fig. 77, and all other vessels used in chemical operations for measuring, are graduated in like manner. The bell glasses, (Fig. 61,) for which and all large vessels, water is preferable, should be graduated into double cubic centimetres, so that every divisional line may correspond to two centimetres; and the tubes into double cubic millimetres, so that every line may correspond to two cubic millimetres.

Dr. Henry proposes, as a quick and accurate method of graduating tubes for eudiometry, &c., to have a standard tube, 0.08 of an inch in diameter, and carefully divided into 10 equal

parts, of 10 grains of mercury ( $60^{\circ}$  F.) capacity each. The vessels should be of clear glass. The tubes must be thick, and strong enough to support the weight of their full contents of mercury. For the convenience of closing their mouths with glass disks, their ends may be ground flat and even.

In all operations of graduation, the waste of mercury is avoided by working over a porcelain plate, or, what is better, the mercury trough, Fig. 79. The metal may be conveyed to the vessels in the pipette, Fig. 59, which enables the addition or removal of minute portions, as the case may require.

The requirements of the laboratory call for an assorted stock of graduated tubes and proof glasses, varying in diameter from a quarter to two inches.

Below is a useful table, showing the value of the measures of capacity in cubic inches, grains, and as compared with apothecaries' measure.

Cubic inches. tilled water. measure.   Imperial gallon 277.274 70000 9.966+   Imperial pint 34.65925 8750   Imperial fluidounce 1.7329625 437.5   The old wine pint 28.8827 7291.666 16 fl. oz.   Old fluidounce 1.805169 455.73 8 drachms			Grains of dis-	Apothecaries'
Imperial gallon 277.274 70000 9.966+   Imperial pint . 34.65925 8750 8750   Imperial fluidounce 1.7329625 437.5 437.5   The old wine pint . 28.8827 7291.666 16 fl. oz.   Old fluidounce 1.805169 455.73 8 drachms		Cubic inches.	tilled water.	measure.
Imperial pint 34.65925 8750   Imperial fluidounce 1.7329625 437.5   The old wine pint 28.8827 7291.666 16 fl. oz.   Old fluidounce 1.805169 455.73 8 drachms	Imperial gallon .	277.274	70000	9.966-
Imperial fluidounce 1.7329625 437.5   The old wine pint 28.8827 7291.666 16 fl. oz.   Old fluidounce 1.805169 455.73 8 drachms	Imperial pint .	34.65925	8750	
The old wine pint 28.8827 7291.666 16 fl. oz.   Old fluidounce 1.805169 455.73 8 drachms	Imperial fluidounce	1.7329625	437.5	
Old fluidounce 1805169 45573 8 drachms	The old wine pint .	28.8827	7291.666	16 fl. oz.
ora naradando . 1.000100 Toorro O aradanis.	Old fluidounce .	1.805169	455.73	8 drachms.
Cubic inch 1. 252.458	Cubic inch	1.	252.458	
Litre 61.02525 15406.312 2.1135 pints.	Litre	61.02525	15406.312	2.1135 pints.
Decilitre 6.10252 1540.631 3.3816 fl. oz.	Decilitre	6.10252	1540.631	3.3816 fl. oz.
Centilitre 0.61025 154.063 2.7053 fl. drachms	Centilitre	0.61025	154.063	2.7053 fl. drachms.
Millilitre 0.06102 15.406 16.2318 minims.	Millilitre	0.06102	15.406	16.2318 minims.

Measurement of Gases .- In measuring, a required volume of any gas, a graduated tube, like the one shown in Fig. 78, is first filled with mercury or water as the case may be, in the pneumatic trough, and placed upon the shelf. When the tube is too slender to sustain itself in an upright position, it is then convenient to use the clamp and support, A Fig. 79. If the mouth of the receptacle of the gas is wide, it is necessary, before transferring to the graduating tube, to place a small funnel in its submerged end, so that the ascending bubbles may be received upon a larger surface. By giving the reservoir, generally a bell glass, a slightly inclined position, so that the edge of its mouth may reach under the funnel, the transfer is made easily and without loss. As soon as the requisite quantity has been transferred, the connection must be broken, and both the bell and tube made to resume their former positions on the shelf. (See Transfer of Gases.) The tube is then to be depressed in the trough until the metal, inside and outside, is at the

#### MEASUREMENT OF GASES.



same level. This mode subjects the gas only to atmospheric pressure, but the tube must be held by a cork-lined clamp, as in Fig. 79, or linen holder, and not in the naked hand, the warmth of which, by expanding the gas, would be a source of error.

It is very difficult to transfer a quantity of gas exactly corresponding with a division of the tube at one trial—several attempts are requisite, except in cases of consummate manipulation. It is perhaps better to transfer the last portions from a small tube. The gas passing through very slowly and in fine bubbles can by this arrangement be stopped off as soon as the volume which has entered accords with the division indicated. When more than sufficient has been transferred, place the first finger upon the mouth of the tube so as to leave a partial opening, and incline it sufficiently to allow the exit of the redundant gas. Examine anew the contained volume, and if it is still in excess, repeat this operation until the level of the liquid reaches the proper height.

To insure accuracy in the comparison of volumes of different gases, they must necessarily be measured at the same temperature and under the same pressure. The proof glasses in which they are estimated should be kept out of the influence of unequal warmth during the process, for the action of heat upon the volume of gases is a cause of considerable error.

In order to determine with precision, the exact height which the water or mercury assumes, the vessel should be placed at repose upon a level shelf, and the eye directed on a line with the surface of the fluids, and the height read off accordingly. This notation requires some care and precision, for as mercury assumes a convex surface, owing to its own cohesion, and water a concave one, because of the attraction for the walls of the tube, especially in narrow cylinders, the curve thus occasioned presents an impediment to the ready determination of the exact level.

When water is the confining fluid, read the real surface in the middle of the dark zone formed by the water around the inner walls of the tube; on the other hand, when mercury is used, "place the real surface in a line drawn exactly in the centre between the highest point of the surface of the mercury and the points at which the latter is in actual contact with the walls of the tube."

In either case the temperature of the fluid and gas should be uniform. When the bulk of the containing fluid is sufficient to allow the entire immersion of the cylinder, this is easily effected; otherwise, it becomes necessary to equalize the temperature of the surrounding air, by keeping the cylinder exposed to both, in order to determine accurately the degree of the scale at which the mercury or water stands.

Another important matter, as before mentioned, in the comparison of volumes of different gases, is the necessity of uniform pressure, in their measurement. If the level of the containing fluid within and without the cylinder exactly corresponds, the pressure upon it is directly shown by the barometer. A higher level, internally, indicates less pressure, and vice versâ: when the fluid stands higher outside of the cylinder than within it, the level may be restored by raising the tube; in the opposite case by depressing the tube. These operations of adjusting the level are more difficult when mercury is the containing fluid. In operations occupying much time, the barometer should be frequently consulted, so as to guard against any alteration sufficient to impair the results.

Fig. 80.

Ker's tube, constructed for the measurement of gas at the time of its disengagement, is shown by Fig. 80. The branch a, ten inches in length, glass stoppered and graduated to two cubic inches, is the recipient of the gas disengaged from the material in the bulb c, by the action of a reagent introduced in the other branch b. The gas collecting in a is there measured by the scale, previous to being transferred for examination.

# CHAPTER X.

### MEASUREMENT OF TEMPERATURE.

TEMPERATURE is estimated by means of two instruments, the *pyrometer* and *thermometer*, the action of which is based upon the relative expansibility of bodies under the influence of heat and cold. They do not therefore indicate the amount of heat contained in a body, but only the comparative temperature of two or more bodies.

The Pyrometer.—This instrument is rarely used in the ordinary operations of the laboratory, it being only applicable to the measurement of heats more intense than can be borne by thermometers. Pyrometers are constructed of solid substances, though gaseous bodies, on account of their sensitiveness to heat or cold and greater uniformity of expansion, would be preferable. Daniell's instrument is the most approved, and by skilful management may be made to give accurate indications. Its principal application is in furnace operations. In assaying, where the required temperature varies with the metal under process, it is particularly available in determining the heat of the furnace; for much of the accuracy of the assay depends upon the temperature at which it is made. Fig. 81 represents the apparatus.

"It consists of two parts, which may be distinguished as the register 1, and the scale 2. The register, A, is a solid bar of black-lead earthenware highly baked. In this a hole, a a, is drilled, into which a bar of any metal, six inches long, may be dropped, and which will then rest upon its solid end.

#### THE PYROMETER.

A cylindrical piece of porcelain c, called the index, is then placed upon the top of the bar, and confined in its place by a ring or strap of platinum d, passing round the top of the



register, which is partly cut away at the top, and tightened by a wedge of porcelain e. When such an arrangement is exposed to a high temperature, it is obvious that the expansion of the metallic bar will force the index forward to the amount of the excess of its expansion over that of the blacklead, and that when again cooled it will be left at the point of greatest elongation. What is now required, is the measurement of the distance which the index has been thrust forward from its first position, and this, though in any case but small, may be effected with great precision by means of the scale."

"This is independent of the register, and consists of two rules of brass, f f and g, accurately joined together at a right angle by their edges, and fitting square upon two sides of the blacklead bar. At one end of this double rule, a small plate of brass h, projects at a right angle, which may be brought down upon the shoulder of the register formed by the notch cut away for the reception of the index. A movable arm D, is attached to this frame, turning at its fixed extremity on a centre i, and at its other carrying the arc of a circle, whose radius is exactly five inches, accurately divided into degrees, and thirds of a degree. Upon this arm, at the centre of the circle k, another lighter arm c is made to turn, one end of which carries a nonius H with it, which moves upon the face 10 of the arc, and subdivides the former graduation into minutes of a degree; the other end crosses the centre and terminates in an obtuse steel point m, turned inwards at a right angle.

"When an observation is to be made, a bar of platinum or malleable iron is placed in the cavity of the register; the index is to be pressed down upon it, and firmly fixed in its place by the platinum strap and porcelain wedge. The scale is then to be applied by carefully adjusting the brass rule to the sides of the register, and fixing it by pressing the cross piece upon the shoulder, and placing the movable arm so that the steel part of the radius may drop into a small cavity made for its reception, and coinciding with the axis of the metallic bar. The minute of the degree must then be noted which the nonius indicates upon the arc. A similar observation must be made after the register has been exposed to the increased temperature which it is designed to measure, and again cooled, and it will be found that the nonius has been moved forward a certain number of degrees or minutes. The scale of this pyrometer is readily connected with that of the thermometer by immersing the register in boiling mercury, whose temperature is as constant as that of boiling water, and has been accurately determined by the thermometer. The amount of expansion for a known number of degrees is thus determined, and the value of all other expansions may be considered as proportionate."

"The following is a list of the melting points of some of the metals, and it is obvious that in an assay of each particular metal, the temperature employed must exceed by a considerable number of degrees its melting point. The table is, therefore, very useful.

						r	anrenneit.
Tin melts	at				· .		$422^{\circ}$
Bismuth							497
Lead							612
Zinc			. ·				773
Cadmium							442
Silver							1860
Copper					· · ·		1996
Gold							2016
Cast iron							2786
Cobalt an	d nick	el r	ather	less	fusible	than	iron."

Daniell.

Thermometers.-A thermometer consists of a graduated cylindrical stem, with a uniform capillary bore, having one of its ends blown into a bulb and filled with mercury or alcohol, and the other hermetically closed, the space above the column of fluid being a vacuum, or as nearly as possible devoid of air.

Mercury, on account of its greater equability of expansion, and of its boiling point being as high as 650° F., is more available in the construction of thermometers for measuring temperatures exceeding that of boiling water (212° F.). Alcohol, on the other hand, by reason of its eminent property of dilatation is more applicable for determining temperatures lower than the freezing point of mercury, its point of congelation being as far down as -90° F.

The two points of graduation are the freezing and boiling points of water, the interval between each being differently apportioned, according as the scale of Fahrenheit, Celsius, or Reaumur (the three most in use) is employed.





Fig. 82.

Fahrenheit's scale ranges from  $32^{\circ}$  to  $212^{\circ}$ ; that of Celsius (centigrade) from  $0^{\circ}$  to  $100^{\circ}$ ; Reaumur's from  $0^{\circ}$  to  $80^{\circ}$ . The first is most popular in England and in this country; the second in France, and the third in Russia, Spain, and part of Germany. The scale of Fahrenheit has its zero at  $32^{\circ}$  below the freezing point of water, and the other two exactly at that point. Therefore, in comparing the degrees of the former with those of the latter, the negative or those below zero have a prefix of the minus (—) sign, and the positive or those above, the plus (+) sign. The diagram (Fig. 83) will present the relative position of the corresponding degrees of the three scales.

The following rules will be found convenient for translating the degrees of one scale into those of another:

1. To reduce Centigrade degrees to those of Fahrenheit, multiply by 9, and divide by 5, and to the quotient add 32, that is,—

$$\frac{\text{Cent.} \times 9}{5} + 32 = \text{Fahr.}$$

3. To reduce Reaumur's to Fahrenheit's:—  $\frac{\text{Reau.} \times 9}{4} + 32 = \text{Fahr.}$ 

4. To convert Fahrenheit's to Reaumur's:—  $\frac{\text{Fahr.} - 32 \times 4}{9} = \text{Reaumur.}$ 

A slender stem and precise uniformity of bore are indispensable to the accuracy of a thermometer. The tube must also be entirely void of air, as is known upon its inversion when the contained mercury makes a free and rapid descent. Moreover, the graduation of the scale must be verified, and to do this, the bulb is immersed in a mixture of salt and snow to test the accuracy of its freezing degree, and afterwards in boiling water (under the ordinary pressure of the atmosphere) to observe its boiling point. If in either case when the fluid becomes stationary, after sufficient delay for the bulb to acquire the temperature of the bath, it corresponds with the degree marked upon the scale, its graduation as regards the freezing and boiling points is correct. To determine the exactness of the intermediate space, the length of the interval is measured with a pair of compasses, and it is then easy to ascertain by means of an accurate ruler, if the divisions accord with each other, and in the aggregate with the total length of the scale.

For measuring temperatures higher than 580° F., the top of the thermometer should be unsealed and the mercury exposed to the pressure of the atmosphere, for if hermetically closed, it will boil at that point and burst the tube.

The tube, as before said, should be as slender as possible, and not too long, otherwise in testing shallow solutions in ebullition, that part of the stem which is above the liquor is exposed to the heat of the rising vapor, and as the expansion to mercury within would be thus estimated with that of the contents of the bulb, the only part heated at the time of graduation, incorrect conclusions would be drawn.

In ascertaining the condition of a liquid with regard to heat or cold, the thermometer is gradually introduced into it, moved around several times so as to produce an equable diffusion of temperature, and after the mercury has become stationary at a certain point, the degree coincident with that point is noted down as the temperature.

The scales of thermometers are most generally graduated upon a wooden slip or support, to which the stem is secured by clamps and screws. In this case, the scale is hinged (Fig. 82) so as to afford convenience in the use of the thermometer for taking the boiling point of solutions without injury to the scale.

Some manufacturers make the thermometers wholly of glass, and etch the scale upon the broad sides of the flat tube, as shown by Fig. 84. This kind is very convenient for passing through tubulures, but is well replaced by those with the scale written upon paper and enclosed with the thermometer stem in a glass tube. These latter are made in the most skilful manner by Greiner & Co., Berlin. Fisher and Heintz, of Philadelphia, also make excellent thermometers.

The scales of the mercurial thermometers are made to range as high as  $600^{\circ}$  F., and for convenience are sometimes graduated on one side of the stem with the Centigrade and on the other with the Fahrenheit scale. Fahrenheit's degrees being small, have the adFig. 84.

vantage over the others of not giving fractional parts, which are inconvenient in calculation. The laboratory should be supplied with two or more of these apparatus.

Air thermometers are sometimes used, and though very delicate, are less convenient than those of mercury and alcohol, and liable to objections which do not attach to the latter.

Leslie's differential thermometer, Fig. 85, which is a modi-



fication of the air thermometer, is now frequently used in researches for determining very small differences in temperature. It consists of an U tube with a hollow bulb blown at each end and closed, so that the fluid within (sulphuric acid, colored with carmine to render it more visible) is entirely free from external atmospheric pressure. This instrument does not exhibit a change of temperature except by the difference between the elasticity of the air in the two bulbs, and therefore indicates only such temperatures as affect one bulb and not the

other. When both bulbs are of equal temperature, the liquid within remains stationary, but so soon as one becomes warmer than the other the fluid recedes to the opposite bulb, and the scale attached to one of the legs is so graduated as to measure the comparative degree of heat thus occasioned.

Melloni's thermo-multiplicator (Müller, p. 541), is another instrument for the indication of changes of temperature.

Another convenient instrument, especially in meteorological observations, is the *thermometrograph*. It is so constructed as to register the maximum and minimum temperatures occurring during an interval, and hence the presence of the operator is not necessary to note them at the moment of their occurrence.

The apparatus which is shown in Fig. 86, consists of a mer-



curial and a spirit thermometer placed horizontally and parallel to each other. A steel pin enclosed in the tube of the former is pushed before the column of mercury when the metal in the bulb expands, but remains fixed when it again recedes on cooling, and thus indicates at that point the maximum temperature which has occurred during any interval.

The corresponding rod, enclosed in the tube of the spirit thermometer, of glass, colored to render it more visible, is not advanced by the expansion of the spirit, but retreats with the column as it contracts to the last point reached by it, and thus registers the minimum of temperature during a certain time, at the degree coincident with its inner end.

When this instrument is to be used, it must be inclined in such a position as to allow the steel rod to descend to the column of mercury, and the glass rod to the end of the spirituous column. The arrangement of the bulbs in opposite positions is with a view to this object. After the rods have reached their proper situations, we may, by placing the instrument horizontally any morning or evening, obtain at the end of the following 24 hours, the maximum and minimum temperature of that interval.

There are some very excellent remarks by Regnault upon the relative advantages of the different modes of measuring temperature, to which the student may advantageously refer.

The translation of his several papers on the subject, is to be found in the *Franklin Institute Journal* for 1848.

The following table shows the corresponding degrees of Fahrenheit's, Reaumur's, and the Centigrade thermometers.

144 THERMOMETRICAL EQUIVALENTS.

Fahren- heit.	Reau- mur.	Centi- grade	Fahren- heit.	Reau- mur.	Centi- grade.	Fahren- heit.	Reau- mur.	Centi- grade.	Fahren- heit.	Reau- mur.	Centi- grade.
600 599	252.4 252	315.5 315	568.4 568	238.4 238.2	298 297.7	538 537.8	224.9 224.8	281.1 281	506.7 506	211	263.7
598	251.5	314.4	567.5	238	297.5	537	224.4	280.5	505.4	210.4	263
597.2	251.2	314	566 6	237.7	297.2	536	224	280	505	210.2	262.7
596.7	251.1	313.7	566	237.3	296.6	534.2	223.2	279	504.5	209.7	262.2
596	250.3	313.3	565.2	237	296.2	534	223.1	278.8	503.6	209.6	262
595.4	250.4	313	565	236.9	296.1	533.7	223 999 6	278.7	503	209.3	261.6
594.5	250.2	312.5	564	236.4	295.5	532.4	222.4	278	502.2	209	261.2
594	249.7	312.2	563	236	295	532	222.2	277.7	501.8	208.8	261
593.6	249.6	312	561 9	235.5	294.4	531.5	222	277.5	501	208.4	260.5
592.2	249.3	311.0	561	235.2	294 293.8	530.6	221.7	277	499	208	259.4
ō92	248.9	311.1	560.7	235	293.7	530	221.3	276.6	498.2	207.2	259
591.8	248.8	311	560	234.6	293.3	529.2	221	276.2	498	207.1	258.8
591	248.4	310.5	559.4	234.4	293	529 528 8	220.9	276.1	497.7	207	258.7
589	247.5	309.4	558.5	234	292.5	528	220.4	275.5	496.4	206.4	258
588.2	247.2	309	558	233.7	292.2	527	220	275	496	206.2	257.7
588	247.1	308.8	557.6	233.6	292	526	219.5	274.4	495.5	206	257.5
587	246.6	308.3	556.2	233	291.2	525.2	219.2	273.8	494.6	205.6	257
586.4	246.4	308	556	232.9	291.1	524.7	219	273.7	494	205.3	256.6
586	246.2	307.7	555.8	232.8	291	524	218.6	273.3	493.2	205	256.2
585	240	307.2	554	232.4	290.5	523	218.4	273	493	204.9	256
584.6	245.6	307	553	231.5	289.4	522.5	218	272.5	492	204.4	255.5
584	245.3	306.6	552.2	231.2	289	522	217.7	272.2	491	204	255
583.2	245	306.2	551 7	231.1	288.8	521.0	217.6	272	490	203.5	254.4
582.8	244.8	306	551	230.6	288.3	520.2	217	271.2	489	203.1	253.8
582	244.4	305.5	550.4	230.4	288	520	216.9	271.1	488.7	203	253.7
581	244	305	550	230.2	287.7	519.8	216.8	271	488	202.6	253.3
579.2	243.5	304.4	549.5	229.7	287.2	518	216.4	270.5	487	202.4	252.7
579	243.1	303.8	548.6	229.6	287	517	215.5	269.4	486.5	202	252.5
578.7	243	303.7	548	229.3	286.6	516.2	215.2	269	486	201.7	252.2
577.4	242.0	303.5	547	228.9	286.1	515.7	215.1	208.0 268.7	485.0	201.0	251.6
577	242.2	302.7	546.8	228.8	286	515	214.6	268.3	484.2	201	251.2
576.5	242	302.5	546	228.4	285.5	514.4	214.4	268	484	200.9	251.1
575.6	241.7	302.2	540	228	280	513.5	214.2	267.7	483.8	200.8	250.5
575	241.3	301.6	543.2	227.2	284	513	213.7	267.2	482	200	250
574.2	241	301.2	543	227.1	283.8	512.6	213.6	267	481	199.5	249.4
573 8	240.9	301.1	542.7	227 226 6	283.7	511 9	213.3	266.6	480.2	199.2	249
573	240.4	300.5	541.4	226.4	283	511	212.9	266.1	479.7	199	248.7
572	240	300	541	226.2	282.7	510.8	212.8	266	479	198.6	248.3
571	239.5	299.4	540.5	226	282.5	510	212.4	265.5	478.4	198.4	248
570.2	239.2	298.8	539.6	225.6	282.2	508	211.5	264.4	477.5	198.2	247.5
569.7	239	298.7	539	225.3	281.6	507.2	211.2	264	477	197.7	247.2
569	238.6	298.3	538.2	225	281.2	507	211.1	263.8	476.6	197.6	247

## THERMOMETRICAL EQUIVALENTS.

en-			en-			-ua			en-		e.
ahronahronahronahronahronahronahronahron	eau	enti	ahr neit.	eau	enti grad	ahr	mur	enti grad	ahr heit.	mur	enti grac
£		0	4			H-					
476	197.3	246.6	444.2	183.2	229	414	169.7	212.2	383	156	195
475.2	197	246.2	444	183.1	228.8	413.6	169.6	212	382	155.5	194.4
474.8	196.8	240.1	443.7	182.6	228.1	413	169.5	211.0	381.2	155.2	194
474	196.4	245.5	442.4	182.4	228	412	168.9	211.1	380.7	155	193.7
473	196	245	442	182.2	227.7	411.8	168.8	211	380	154.6	193.3
472	195.5	244.4	441.5	182	227.5	411	168.4	210.5	379.4	154.4	193
471	195.1	243.8	440.6	181.6	227	409	167.5	209.4	378.5	154.2	192.5
470.7	195	243.7	440	181.3	226.6	408.2	167.2	209	378	153.7	192.2
470	194.6	243.3	439.2	181	226 2	408	167.1	208.8	377.6	153.6	192
469.4	194.4	243	439	180.9	226.1	407.7	167	208.7	3776 9	153.3	191.0
468.5	194.2	242.5	438	180.4	225.5	406.4	166.4	208.5	376	152.9	191.1
468	193.7	242.2	437	180	225	406	166.2	207.7	375.8	152.8	191
467.6	193.6	242	436	179.5	224.4	405.5	166	207.5	375	152.4	190.5
467	193.3	241.6	435.2	179.2	224	400	165.7	207.2	374	152	190
466	192.9	241.1	434.7	179	223.7	404.0	165.3	206.6	372.2	151.2	189
465.8	192.8	241	434	178.6	223.3	403.2	165	206.2	372	151.1	188.8
465	192.4	240.5	433.4	178.4	223	403	164.9	206.1	371.7	151	188.7
404	192	240	433	178.2	222.7	402.8	164.8	206	371	150.6	188.3
462.2	191.2	239	432	177.7	222.2	401	164.4	205.0	370	150.2	187.7
462	191.1	238.8	431.6	177.6	222	400	163.5	204.4	369.5	150	187.5
461.7	191	238.7	431	177.3	221.6	399.2	163.2	204	369	149.7	187.2
401	190.6	238.3	430.2	176 0	221.2	399	163.1	203.8	368.0	149.0	187
460	190.2	237.7	429.8	176.8	221	398	162.6	203.3	367.2	149	186.2
459.5	190	237.5	429	176.4	220.5	397.4	162.4	203	367	148.9	186.1
459	189.7	237.2	428	176	220	397	162.2	202.7	366.8	148.8	186
408.0	189.6	237	427	175.9	219.4	396.0	162	202.5	365	148.4	185.5
457.2	189	236.2	426	175.1	218.8	395.6	161.6	202.2	364	147.5	184.4
457	188.9	236.1	425.7	175	218.7	395	161.3	201.6	363.2	147.2	184
456.8	188.8	236	425	174.6	218.3	394.2	161	201.2	363	147.1	183.8
400 455	188.4	230.0	424.4	174.4	218	394	160.9	201.1	362.7	147	183.3
454	187.5	234.4	423.5	174	217.5	393	160.4	200.5	361.4	146.4	183
453.2	187.2	234	423	173.7	217.2	392	160	200	361	146.2	182.7
453	187.1	233.8	422.6	173.6	217	391	159.5	199.4	360.5	146	182.5
452.7	186 6	233.7	422	173.3	216.0	390.2	159.2	199	359 6	145.6	182.2
451.4	186.4	233	421	172.9	216.1	389.7	159	198.7	359	145.3	181.6
451	186.2	232.7	420.8	172.8	216	389	158.6	198.3	358.2	145	181.2
450.5	186	232.5	420	172.4	215.5	388.4	158.4	198	358	144.9	181.1
449.6	185.6	232.2	419	171.5	214.4	387.5	158.2	197.5	357	144.8	180.5
449	185.3	231.6	417.2	171.2	214	387	157.7	197.2	356	144	180
448.2	185	231.2	417	171.1	213.8	386.6	157.6	197	355	143.5	179.4
448	184.9	231.1	416.7	171	213.7	386	157.3	196.6	354.2	143.2	179
447	184.4	230.5	415.4	170.4	213.5	385	156.9	196.1	353.7	143.1	178.7
446	184	230.	415	170.2	212.7	384.8	156.8	196	353	142.6	178.3
445	183.5	229.4	414.5	170	212.5	384 -	156.4	195.5	352 4	142.4	178 .

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THERMOMETRICAL EQUIVALENTS.

Fahren- heit.	Reau- mur.	Centi- grade.									
352	142.2	177.7	321.8	128.8	161	290	114.6	143.3	259.2	101	126.2
351.5	142	177.5	321	128.4	160.5	289.4	114.4	143	259	100.8	126.1
351 6	141.8	177.2	320	128	-160 159 4	289	114.2	142.7	258.8	100.8	126
350.0	141.3	176.6	318.2	127.2	159.4	288	113.7	142.0	257	100.4	125.5
349.2	141	176.2	318	127.1	158.8	287.6	113.6	142	256	99.5	124.4
349	140.9	176.1	317.7	127	158.7	287	113.3	141.6	255.2	99.2	124
348.8	140.8	176	317	126.6	158.3	286.2	113	141.2	255	99.1	123.8
340	140.4	175.0	310.4	120.4	157.7	280	112.8	141.1	254.7	99	123.7
346	139.5	174.4	315.5	126.2	157.5	285	112.4	140.5	253.4	98.4	123.5
345.2	139.2	174	315	125.7	157.2	284	112	140	253	98.2	122.7
345	139.1	173.8	314.6	125.6	157	283	111.5	139.4	252.5	98	122.5
344.7	139	173.7	314	125.3	156.6	282.2	1111.2	139	252	97.9	122.2
343.4	138.4	173.0	313.2	120	156.1	281 7	111.1	138.9	251.0	97.0	121 6
343	138.2	172.7	312.8	124.8	156	281	110.6	138.3	250.2	97	121.2
342.5	138	172.5	312	124.5	155.5	280.4	110.4	138	250	96.9	121.1
342	137.7	172.2	311	124	155	280	110.2	137.7	249.8	96.8	121
341.0	137.6	172	310	123.5	154.4	279.5	110	137.5	249	96.4	120.5
340.2	137.3	171.0	309.2	123.2	153.8	278 6	109.7	137.2	240	95.5	119.4
340	136.9	171.1	308.7	123	153.7	278	109.3	136.6	246.2	95.2	119
339.8	136.8	171	308	122.6	153.3	277.2	109	136.2	246	95.1	118.9
339	136.4	170.5	307.4	122.4	153	277	108.8	136.1	245.7	95	118.7
338	136	170	307	122.2	152.7	276.8	108.8	136	245	94.6	118.3
336.2	135.2	169.4	306.5	122	152.2	275	108.4	135.0	244.4	94.2	117.8
336	135.1	168.8	305.6	121.6	152	274	107.5	134.4	243.5	94	117.5
335.7	135	168.7	305	121.3	151.6	273.2	107.2	134	243	93.8	117.2
335	134.6	168.3	304.2	121	151.2	273	107.1	133.8	242.6	93.6	117
334.4	134.4	167 7	304	120.9	151.1	272.7	107	133.7	242	93.3	116.0
333.5	134.2	167.5	303.8	120.8	150.5	271.4	106.4	133.5	241.2	92.9	116.1
333	133.7	167.2	302	120	150	271	106.2	132.7	240.8	92.8	116
332.6	133.6	167	301	119.5	149.4	270.5	106	132.5	240	92.4	115.5
332	133.3	166.6	300.2	119.2	149	270	105.7	132.2	239	92	115
331	133	166.1	300	119.1	148.9	209.0	105.0	132	238	91.0	114.4
330.8	132.8	166	299	118.6	148.3	268.2	105.5	131.2	237	91.1	113.9
330	132.4	165.5	298.4	118.4	148	268	104.8	131.1	236.7	91	113.7
329	132	165	298	118.2	147.7	267.8	104.8	131	236	90.3	113.3
328	131.5	164.4	297.5	118	147.5	267	104.4	130.5	235.4	90.4	113
327	131.2	163.9	296 6	117.6	147.2	265	104	120 4	230	90.2	112.5
326.7	131	163.7	296	117.3	146.6	264.2	103.2	129	234	89.7	112.2
326	130.6	163.3	295.2	117	146.2	264	103.1	128.9	233.6	89.6	112
325.4	130.4	163	295	116.9	146.1	263.7	103	128.7	233	89.3	111.6
320	130.2	162.7	294.8	116.8	146	263	102.6	128.3	232.2	89	111.2
324	129.7	162.2	293	116.4	145.5	262.4	102.4	127.7	231.8	88.8	111
323.6	129.6	162	292	115.5	144.4	261.5	102	127.5	231	88.4	110.5
323	129.3	161.6	291.2	115.2	144	261	101.7	127.2	230	88	110
322.2	129	161.2	291	115.1	143.8	260.6	101.6	127	229	87.5	109.4
344	120.0	101.1	1290.1	110	143.7	200	101.3	120.0	223.21	01.2	109

# THERMOMETRICAL EQUIVALENTS.

ahren- ieit.	eau- aur.	enti- rade.	ahren- ieit	eau- aur.	enti- rade.	ahren- ieit.	eau- nur.	enti- rade.	ahren- eit.	eau- nur.	enti- ade.
E	8.	0° C	F	R.	<sup>00</sup> ت	<u>E</u>	2 a	0° 00	P.P.	Ra	0.60
228	87.1	108.9	197.6	73.6	92	166	59.5	74.4	135.5	46	57.5
227.7	87 86 6	108.7	197	73.3	91.6 91.2	165.2	59.2 59.1	74 73 9	135	45.6	57.2
226.4	86.4	108	196	72.8	91.1	164.7	59	73.7	134	45.3	56.6
226	86.2	107.8	195.8	72.8	91	164	58.6	73.3	133.2	45	56.2
220.0	85.7	107.5	195	72.4	90.5	163.4	58.2	72.7	133	44.9	56
224.6	85.6	107	193	71.5	89.4	162.5	58	72.5	132	44.5	55.5
224	85.3	106.6	192.2	71.2	89	162	57.7	72.2	131	44	55
223.2	84 9	106.2	192	71.1	88.7	161.0	57.3	72	130	43.0	54.4
222.8	84.8	106	191	70.6	88.3	160.2	57	71.2	129	43.1	: 53.9
222	84.4	105.5	190.4	70.4	88	160	56.8	71.1	128.7	43	53.7
221	84	105	190	70.2	87.8	159.8	56.8	71	128	42.6	53.3
219.2	83.2	104.4	189.5	69.7	87.2	158	56	70	127.4	42.2	52.7
219	83.1	103.9	188.6	69.6	87	157	55.5	69.4	126.5	42	52.5
218.7	83	103.7	188	69.3	86.6	156.2	55.2	69	126	41.8	52.2
218	82.0	103.3	187.2	68.9	80.2	155.7	55.1	68.7	125.0	41.0	51.6
217	82.2	102.7	186.8	68.8	86	155	54.6	68.3	124.2	41	51.2
216.5	82	102.5	186	68.4	85.5	154.4	54.4	68	124	40.9	51.1
216	81.7	102.2	185	67 5	80	153 5	54.2	67.7	123.8	40.8	50 5
215	81.3	101.6	183.2	67.2	84	153	53.7	67.2	122	40.1	50
214.2	81	101.2	183	67.1	83.9	152.6	53.6	67	121	39.5	49.4
214	80.9	101.1	182.7	67	83.7	152	53.3	66.6	120.2	39.2	49
213.0	80.4	100.5	182	66.4	83	151.4	52.9	66.1	119.7	39.1	48.7
212	80	100	181	66.2	82.7	150.8	52.8	66	119	38.6	48.3
211	79.5	99.4	180.5	66	82.5	150	52.4	65.5	118.4	38.4	48
210.2	79.2	99	180	65.6	82.2	149	51 5	64 4	117.5	38.2	47.5
209.7	79	98.7	179	65.3	81.6	147.2	51.2	64	117	37.7	47.2
209	78.6	98.3	178.2	65	81.2	147	51.1	63.9	116.6	37.6	47
208.4	78.4	98.0	178	64.9	81.1	146.7	50 6	63.7	116	37.3	46.0
207.5	78	97.5	177	64.4	80.5	145.4	50.4	63	115	36.9	46.1
207	77.7	97.2	176	64	80	145	50.2	62.7	114.8	36.8	46
206.6	77.6	97	175	63.5	79.4	144.5	50	62.5	114	36.4	45.5
205.2	77	96.0	174.2	63.1	78.8	143.6	49.6	62	112	35.5	44.4
205	76.9	96.1	173.7	63	78.7	143	49.3	61.6	111.2	35.2	44
204.8	76.8	96	173	62.6	78.3	142.2	49	61.2	111	35.1	43.9
204	76.4	95.5	172.4	62.2	77.7	141.8	48.9	61	110.7	30	43.7
202	75.5	94.4	171.5	62	77.5	141	48.4	60.5	109.4	34.4	43
201.2	75.2	94	171	61.7	77.2	140	48	60	109	34.2	42.7
201	75.1	93.9	170.6	61.6	76 6	139	47.0	59.4	108.5	34	42.5
200.1	74.6	93.3	169.2	61	76.2	138	47.1	58.8	107.6	33.6	42
199.4	74.4	93	169	60.8	76.1	137.7	47	58.7	107	33.3	41.6
199	74.2	92.7	168.8	60.8	76	137	46.6	58.3	106.2	33	41.2
198.5	73.7	92.5	167	60.4	75	136.4	46.2	57.7	105.8	32.9	41.1

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THERMOMETRICAL EQUIVALENTS.

Fahren- heit.	Reau- mur.	Centi- grade.	Fahren- heit.	Reau- mur.	Centi- grade.	Fahren- heit.	Reau- mur.	Centi- grade.	Fahren- heit.	Reau- mur.	Centi grade.
105 104	32.4 32	40.5 40	73.4 73	18.4 18.2	23 22.7	43 42.8	4.9 4.8	6.1 6	11.7	- 9 - 9.3	-11.2 -11.6
103 102.2	31.5 31.2	39.4 39	72.5	18 17.7	22.5	42	4.4	5.5 5	10.4	-9.6 -9.7	-12 -12.2
102	31.1	38.9	71.6	17.6	22	40	3.5	4.4	9.5	-10	-12.5
101.7	30.6	38.3	70.2	17.5	21.0	39	3.1	3.9	8.6	-10.2 -10.4	-13
100.4	30.4	38	70	16.9	21.1	38.7	3	3.7	8	-10.6	-13.3
99.5	30.2 30	37.5	69.8 69	16.8	20.5	38	2.0	3.3	7	-11 -11.1	-13.7 -13.9
99	29.7	37.2	68	16	20	37	2.2	2.7	6.8	-11.2	-14
98.6 98	29.6	37 36.6	66.2	15.5	19.4	36.5	2	2.5	6 5	-11.5 -12	-14.4 -15
97.2	29	36.2	66	15.1	18.8	35.6	1.6	2	4	-12.4	-15.5
97	28.9	36.1	65.7	15	18.7	35	1.3	1.6	3.2	-12.8	-16
96	28.4	35.5	64.4	14.4	18	34	0.9	1.1	2.7	-13	-16.2
95	28	35	64	14.2	17.7	33.8	0.8	1	2	-13.3	-16.6
94 93.2	27.5	34.4	63.5	14 13.7	17.5	32	0.4	0.5	1.4	-13.0 -13.7	-17.2
93	27.1	33.9	62.6	13.6	17	31	-0.4	- 0.5	0.5	-14	-17.5
92.7 92	27 26.6	33.7	62 61.2	13.3	16.6	30.2	-0.8 -0.9	-1 -1.1	- 0.4	-14.2 -14.4	-17.7
91.4	26.4	33	61	12.9	16.1	29.7	-1	- 1.2	- 1	-14.6	-18.3
91	26.2	32.7	60.8	12.8	16	29	-1.3	-1.6	$-\frac{1.7}{2}$	-15	-18.7 -18.9
90	25.7	32.2	59	12.4	15.5	28	_1.7	- 2.2	- 2.2	-15.2	-19
89.6	25.6	32	58	11.5	14.4	27.5	-2	- 2.5	- 3	-15.5	-19.4
89 88.2	20.3 25	31.0	57.2	11.2	14	26.6	-2.2 -2.4	- 2.7	$-\frac{4}{5}$	$-10 \\ -16.4$	-20 -20.5
88	24.9	31.1	56.7	11	13.7	26	-2.6	- 3.3	- 5.8	-16.8	-21
87.8	24.8	31	56	10.6	13.3	25.2	-3	- 3.7	-6	-16.8	-21.1 -21.2
.86	24	30	55	10.2	12.7	24.8	-3.2	- 4	- 7	-17.3	-21.6
85	23.5	29.4	54.5	10	12.5	24	-3.5	- 4.4	- 7.6	-17.6	-22
84	23.2	28.9	53.6	9.6	12.2	22	-4.4	- 5.5	- 8.5	-18	-22.5
83.7	23	28.7	53	9.3	11.6	21.2	-4.8	- 6	- 9	-18.2	-22.7
82.4	22.0 22.4	28.3	52.2	9 8.9	11.2	20.7	-4.9	-6.2	-10	-18.4 -18.6	-23.3
82	22.2	27.7	51.8	8.8	11	20	5.3	- 6.6	-10.7	-19	-23.7
81.5	22 21.7	27.5	51	8.4	10.5	19.4		-7 -7.2	-11 -11.2	-19.1 -19.2	-23.8 -24
80.6	21.6	27	49	7.5	9.4	18.5	-6	- 7.5	-12	-19.5	-24.4
80	21.3	26.6	48.2	7.2	9	18	-6.2	- 7.7	-13	-20 -204	-25 -255
79	20.9	26.1	40 47.7	7	8.7	17	-6.6	- 8.3	-14.8	-20.4 -20.8	-26
78.8	20.8	26	47	6.6	8,3	16.2	-7	- 8.7	-15	-20.9	-26.1
77	20.4	25.5	40.4	0.4 6.2	8	15.8	-7.2	- 9	-15.2 -16	-21 -21.3	-26.2 -26.6
76	19.5	24.4	45.5	6	7.5	15	-7.5	- 9.4	-16.6	-21.6	-27
75.2	19.2	24 23.8	45	5.7	7.2	14	-8.4	-10 -10.5	-17 -17.5	-21.7 -22	-27.2 -27.5
74.7	19	23.7	44	5.3	6.6	12.2	-8.8	-11	-18	-22.2	-27.7
74	18.6	23.3	43.2	5	6.2	12	-8.9	-11.1	-18.4	-22.4	-28

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SOURCES AND MANAGEMENT OF HEAT.

Fahren-	Reau- mur.	Centi- grade.	Fahren- heit.	Reau- mur.	Centi- grade.	Fahren- heit.	Reau- mur.	. Centi- grade.	Fahren- heit.	Reau- mur.	Centi- grade.
-19	-22.6	-28.3	-25	-25.3	-31.6	-30	-27.5	-34.4	-35.5	-30	-37.5
-19.7	-23	-28.7	-25.6	-25.6		-31	-28	-35	-36	-30.2	-37.7
-20	-23.1	-28.9	-26	-25.7	-32.2	-32	-28.4	-35.5	-36.4		-38
-20.2	-23.2	-29	-26.5	-26	-32.5	-32.8	-28.8	-36	-37	-30.6	-38.3
-21	-23.5	-29.4	-27	-26.2	-32.7	-33	-28.9	-36.1	-37.7	31	-38.7
-22	-24		-27.4	-26.4		-33.2	-29	-36.2	-38	-31.1	-38.9
-23	-24.4		-28	-26.6	-33.3	-34	-29.3	-36.6	-38.2	-31.2	-39
-23.8	-24.8	-31	-28.7	-27	-33.7	-34.6	-29.6	-37	-39	-31.5	
-24	-24.9	-31.1	-29	-27.1		-35	-29.7	-37.2	-40		-40
-24.2	25	31.2	-29.2	-27.2	-34						

## CHAPTER XI.

#### SOURCES AND MANAGEMENT OF HEAT.

HEAT plays an important part in changing the state and properties of bodies, and we, therefore, devote a chapter to the various modes of applying that agent in chemical operations. The processes dependent upon its action are, principally, FUSION, IGNITION, CALCINATION, INCINERATION, ROAST-ING, DEFLAGRATION, REDUCTION, CUPELLATION, SUBLIMATION, DISTILLATION, DIGESTION, DECOCTION, BOILING, SOLUTION, EVAPORATION, CRYSTALLIZATION, and DESICCATION.

FURNACES.—Laboratory furnaces differ in construction according to the uses for which they are designed. The main parts of every furnace are the body in which the heat is produced, the grate or bars upon which the fuel rests, the ash pan for receiving the residue, and smoke pipe for conducting off the gaseous products of combustion.

The stationary furnace, Fig. 6, answers very well for general purposes, but is less convenient in a small laboratory than a portable furnace. When the former is not possessed by the chemist, the sand bath may be constructed as directed at p. 56, or in default of gas as a heating medium, the top of the stove (p. 39) may serve as a substitute. Such an arrangement is cleanly and economical, and does away with the necessity of cumbersome brick work. It has also the advantage of being ready at all times for use, and is all-sufficient for the purposes of analysis in a private experimental laboratory.

It is useless to multiply furnaces in a small laboratory, for



they occupy room which may be wanting for other purposes, and therefore, a selection should be made of one which in its arrangement is applicable to all the necessities of the chemist. Of this kind, Luhmè's and Kent's are the best. One of either, with a small charcoal furnace, Fig. 87, such as may be bought at any crockery shop, for table use, will constitute the whole stock required of this sort of apparatus.

Luhne's furnace.—Figs. 88, 89 exhibit this furnace, the cylindrical form of which is to be preferred on account of its producing a higher heat with less fuel than any other. It is of strong plate-iron, and lined in the body and dome with refractory fire clay. Its dimensions are twenty-four



inches in height, and nine inches in diameter. The body, a, b, c, d, is capped with a ring of the same circumference as the clay cylinder beneath. The doors are shown at g and h. The circular openings, x, x, opposite to each other, are for the passage of tubes, and when out of use can be closed by the plugs accompanying the furnace for that purpose. The interior of the furnace, as seen from above, is shown by Fig. 90. The knobs e, e, e, projecting inwardly, serve as supports for vessels which are smaller than the mouth of the furnace,

### LUHME'S UNIVERSAL FURNACE.

whilst the iron juts,  $d \ d$ , directed outwardly, are rests for the larger, this arrangement being necessary in both instances, to the perfection of the draught. The iron jacket, Fig. 91,



adapted to the opening, a d, Fig. 88, forms a support for the double sand bath, Fig. 92, for retorts, and other glass vessels. The slope on the side of the sand bath is for the exit of the neck of the retort; and the circular openings, k k k k, Fig. 93, are fitted with covers, by which to augment or decrease the draught, as may be required.

A supplementary sand bath, Fig. 94, is made with a broad extent of surface for digestions, evaporations, &c.

Fig. 93.

Fig. 94.



The dome, Fig. 95, confers the power of a wind furnace when high heat is required. As this chimney becomes too hot to be handled, it is removed when heated with suitable tongs, the form of which is shown in Fig. 96.

The relative position of the several parts of this furnace, we give in the annexed drawing. Fig. 97.

Kent's universal furnace, which is an improvement upon the above, is shown by Fig. 98 in front and side views at A and B. The body is fourteen inches high, by seven inches in diameter, and in material and general construction is similar to Luhmè's furnace. There are six doors:—one at the base for the admission of air, another in the middle for the entrance of the fuel and for the reception of the muffle used in cupellation. The door in the dome is for the purpose of feeding the fire in crucible operations; and that in the side, at the top, for the reception of the neck of a retort, or of the sand bath c, Fig. 98. The two lateral openings, opposite to each other, are for the passage of tubes, or of an iron bar as a support to the rear end of the muffle.





Fig. 97.

Fig. 96.

The two sand baths for distillation and evaporation are seen at c and D, Fig. 98.

The plugs E are for closing the two circular openings by which it is coupled with the pipes connecting it with the laboratory flue. In crucible operations, the smoke-pipe should lead from the top opening, and in evaporations, from the aperture in the back. The openings in the flue must be above the level of the furnace.

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The remaining opening at the base is for the introduction of the mouth of a bellows, by which it may be converted into a blast furnace.

As our advice is to select a single furnace, combining in its construction the power and convenience of the several different kinds required in the laboratory, we proceed to make known how Luhmè's or Kent's apparatus may be adapted to the various purposes of the chemist.

1. As an evaporating and calcining furnace.—As very high heat is seldom required for evaporations, the body of the furnace alone answers every purpose. For small operations, or when but a small fire is required, its capacity may be diminished by inserting an inner cylinder of baked clay. To increase the draught, all the doors should be closed, and to augment still further the heat, as is necessary in the calcination of certain substances, the dome and chimney may be used. In this latter case, by means of the door in the middle, the progress of the operation may be examined without removing the chimney dome, or cooling the interior of the furnace.

The sand and other baths, which have their places upon the top of this furnace, serve for digestions, evaporations, &c., in vessels which require the abatement and equalization of the heat by intermedia.

2. As a reverberatory furnace.—This kind of furnace is adapted to operations demanding a high temperature, as in the heating of crucibles, tubes, &c., and also in sublimation and similar processes requiring the application of a steady heat to all portions of the vessel, rather than a very great heat to any one part of it.

Luhme's or Kent's furnace is rendered reverberatory by the use of the dome, which allows the vessel to be entirely surrounded by flame, and reflects back the heat upon and around its whole surface, and thus by equalizing the temperature, prevents the condensation of vapors in the upper parts, an important object in distilling from beaked vessels.

Coke or charcoal is the fuel generally used, the latter being preferable for a furnace of small dimensions; and the draught may be increased by lengthening the chimney.

The crucible, or vessel, must be placed in the centre, supported upon half of a fire-brick, in such a situation that the cold air ascending through the grate may not prevent the heating of its bottom. The fire is then kindled and maintained by fresh supplies of fuel, which are added carefully so that they may not, whilst cold, come in contact with the hot vessel and occasion its fracture.

3. As a wind furnace.—Wind furnaces are used for the vitrification of mixtures, reduction and fusion of metals, and for other operations requiring a prolonged elevation of temperature.

The combustion is urged by the draught of a flue, and the degree of heat within the furnace depends upon the size and height of the chimney into which this flue passes. The intensity of the heat is increased by so proportioning the dimensions of the furnace and the chimney that their diameters are equal, and the height of the latter twenty to thirty times the diameter of the former.

Luhmè's or Kent's furnace may be converted into a wind furnace by putting on the dome, closing all the openings, and giving a free access of air to the grating through a pipe attached to the circular nozzle in the hearth space, and leading into one of the flues of the laboratory chimney. The smokepipe may lead into the same flue, and both should be fitted with dampers for the regulation of the draught.

4. As a blast furnace.—Blast furnaces are serviceable for expeditiously producing a great intensity of heat, and are used for fusions and other operations which require more power than that of the wind furnace.

The combustion is urged by a current of air forced through

a pair of double bellows, the nozzle of which leads into the circular opening near the base of either of the aforenamed furnaces. The connection should be tightly adjusted with LUTE, so as to prevent any escape of air. The arrangement otherwise is exactly the same as for the wind furnace.

In blowing the blast, let the stream of air entering the furnace be small at first, and be gradually increased as the temperature becomes higher. The maximum heat can be hastened by weighting down the bellows, and thus augmenting the force of the blast.

Sefstrom's (*Berzelius*, vol. 8), and Aikin's (*Faraday*, p. 95), blast furnaces are said to give heat sufficient to melt felspar.

The blast may be furnished to the preceding furnaces from the pneumatic table, Fig. 30, through a flexible leaden pipe, connected at either end by means of coupling screws. As the lead pipe might be softened by a too great proximity to the heated furnace, the opening in the ash pit of the latter to which the former is to be attached, should be fitted with about two feet of iron gas pipe so as to prevent direct contact.

5. As an assay or cupel furnace.—The same arrangement

which is directed for a reverberatory will convert Luhmè's or Kent's into a cupel furnace; the only additional requisite being a muffle, Fig. 99, for the reception of the cupels in assaying operations.

A very convenient and effective furnace for CUPELLATION, is shown in views by Figs. 100, 101.

It is made of refractory fire clay, and hooped with strong iron bands fastened together by screws in order that it may better withstand the high temperature to which it is subjected.

A, A', is the ash-pan, of diameter sufficient for the reception of the body of the furnace B B'. The door, c, is for the exit of the cinders, and the ingress of the air. The larger opening, D', in the body of the furnace, is for the introduction of the muffle, and a corresponding one, D, opposite, for a prismshaped support of baked clay for maintaining the muffle in a horizontal position. The mouth-piece, supported by a small platform, affords the facility of admitting or preventing the access of air to the interior of the muffle.

There are other openings throughout the circumference of

Fig. 99.



## LIEBIG'S FURNACE.

the body immediately above the grate, for increasing the draught when necessary.



In the part of the dome E is a door for the introduction of the fuel. The two openings  $e \ e$  are for the introduction of a poker to arrange the fire.

At the top of the furnace is a dome G G, to which is adapted a sheet iron pipe for increasing the draught.

A sliding door H and a small circular gallery i, as a support for heated coals, afford additional means of increasing the draught.

Furnaces of this kind may be had at the pottery of Haig & Co., or of A. Miller, of this city, reference being given to the form and directions in this book.

Liebig's furnace.—This is a small sheet iron furnace with movable partitions and screen, in which the combustion of or-

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#### MANAGEMENT OF FURNACES.

ganic bodies is effected by a charcoal fire. interior. Fig. 103 gives a side view of the furnace containing a combustion tube under process connected with organic analysis. It is twenty-four inches in length, three inches in height, and three inches in



Fig. 102 shows its

width at the bottom, diverging to four inches at the top.



The combustion tube a passes through a circular opening in the closed end of the furnace and rests upon sheet iron supports, Fig. 104. The grate consists of a

series of slits in the bottom of the furnace which are distant from each other about half an inch. The sheet iron screens, Fig. 105, are used to confine the fire to certain parts of the tube.

The furnace is used upon the table,

and should rest upon a stone of length nearly equal to its own. Management of furnaces.—All furnace operations should be conducted under the stationary hood, Fig. 9, so that the carbonic acid and other noxious exhalations may have an escape from the laboratory, and the sparks and heated air emitted, be prevented from endangering the comfort and safety of the apartment.

If the furnace is without feet, it should rest upon a stone block, and never directly upon the floor or the top of the table, for its heated bottom may occasion a conflagration.

Coal, coke and charcoal are the fuel most used. Coal is the least available, for it contains sulphur, and yields a large amount of ash and clinker, which choke the grating, and it should never, therefore, be used in the blast furnace.

Coke and charcoal, separately or combined, are used for all the furnace operations, the former being preferable for



assays at a high temperature. Weight for weight, their amount of heat is nearly equal, but the greater density of the coke enables it to give more bulk for bulk by ten per cent. Charcoal ignites most readily, but coke is more durable. Moreover, when of good quality and free from sulphurous and earthy matter, it gives but little ash or clinker. By mixing the two together we obtain the good qualities of both; but charcoal alone is preferable for heating glass and porcelain vessels. Before using the coke or charcoal, care must be taken that it has been freed from dust and dirt by sieving, and that the pieces are about the size of a walnut, so that they may pack away neither too loosely nor too compactly.

All of the fuel should be kept in a dry place, for the vapor arising from wet coal and condensing upon the surface of fragile vessels which are being heated, will be apt to cause their fracture.

The crucibles should be placed in the centre of the furnace,



upon a support which may be a piece of fire-brick or a cast-iron trivet, as shown by Fig. 106. This support answers also for stone-ware retorts; but a preferable form for this purpose is the crow's-foot, Fig. 107. The size of these latter imple-

ments is regulated by the proportions of the vessels which they support.

For supporting basins and flasks over the evaporating fur-

Fig. 108.



nace, an iron trellis of strong wire, Fig. 108 is necessary. A series of these iron trellises, of different sized meshes, will be found convenient for adapting the heat to glass vessels, tubes, &c.

In placing the vessels in the fire or in the sand bath, they must be made to stand firmly, and as near to the centre as possible, so that they may be equally heated all around. To prevent damage to them by a too sudden rise of temperature, the fire must be

urged gradually, and when the operations are finished, they should be left to cool with the furnace, or, if taken out, be transferred to a cool sand bath, so that their refrigeration may not be so sudden as to cause fracture.

#### THE FURNITURE OF FURNACES.

When the vessel, to be heated over the naked fire, is of less diameter than the mouth of the furnace, this latter may be proportionally lessened by means of a suitably adapted flat iron ring. These rings Figs. 109, 110, are also useful when it is



required to concentrate the heat of the furnace in the centre of the vessel, and therefore it is advisable to have a series of them, the centre openings of which should decrease gradually so as to render them convenient for all sized vessels.

Before commencing operations the furnace must be entirely

freed from ashes and clinker, and the coal placed around the vessel in layers.— When a fresh supply of fuel is requisite, it may be added through the doorway made for the purpose. The auxiliary apparatus of a furnace, other than that already mentioned, are an ordinary iron poker for clearing the



grate; a pair of tongs bent at right angles, Fig. 111, for placing the crucibles in the fire, and another pair curved at their ends for grasping the crucibles around the body and removing them from the furnace, if necessary, whilst still hot. Another pair of common fire tongs, Fig. 113, is convenient for adding the lumps of coal.

Fig. 113.

LAMPS.—Lamps are convenient and economical substitutes for furnaces in table operations. Being less cumbersome and more cleanly than the latter, they are readily manageable and always ready for use; and they also afford the means of more rapidly multiplying results.

The amount of heat to be obtained by these instruments depends upon their size and arrangement. A properly constructed lamp may be made subservient to all the requirements of the nicer heating operations of the laboratory, from gentle digestion or evaporation to those processes which require a very high degree of heat.

The heating power of the flame is most active immediately beneath its summit, and the vessel should be gradually brought into direct contact with that portion. The vessel should be heated more gradually in proportion to the thickness. When thick glass or porcelain or other fragile bad conducting material is suddenly heated, the heated part expands while the rest does not, and this unequal tension of two adjacent parts causes the cracking or fracture of the vessel. There is, therefore, a great advantage in employing glass or porcelain vessels of thin structure, for the heat being rapidly conducted through them, the liability of fracture is diminished. As strength is, however, often required and thicker vessels must be used, the above principles of expansion and conduction must be remembered when they are employed.

In order to apply a small fire to a large surface, the heat may be diffused by setting the vessel in a SAND or WATER-BATH, or, which is convenient and more cleanly, a plate of sheet metal or wire gauze may be placed between the vessel and the fire. It is safer not to allow the vessel to touch the plate or gauze. Iron or brass gauze may be used, although fine copper gauze is preferable, because more durable.

The combustible or fuel most commonly used in chemical lamps is alcohol, though pyroxylic spirit and lamp oil are occasionally employed.

Alcohol flame gives no smoke or unpleasant odor, the product of combustion being only carbonic acid and water; while lamp oil, especially where the supply of oil to the wick is insufficient, produces a black carbonaceous deposit upon the bottom of the vessel which occasions a loss of heat by radiation.

The alcohol flame moreover does not have the same inju-

rious effect upon bodies in contact with it as the oil flame with its sooty deposit; nor does it hide from view the contents of test-tubes, retorts and other vessels by blackening the glass.

A strong heat may be obtained from alcohol, but in tedious processes, which require a long-continued uniformity of temperature, the *best* lamp oil, or *better*, olive oil may be used in an Argand burner.

Pyroxylic spirit is less objectionable than lamp oil, and more so than alcohol. The many advantages of the latter, therefore, give it the preference over all other combustibles as fuel for chemical lamps. It should be of about the sp. gr. of 0.85. Lamps burning, should always be extinguished before having the supply of fuel renewed, so as to prevent liability of explosion. The spirit is then gradually introduced from the tubed bottle, Fig. 38, p. 72, until the reservoir is nearly full. This mode prevents its running out and diminishes the risk of overflow from too large a stream. When the lamp is not in use, the wick should always be covered with the extinguisher to prevent loss by evaporation.

The tongs accompanying these lamps are a pair of surgeons' forceps of such a form as shown by Fig. 114. As they are liable to become oxidized by constant exposure, it is better to have their prongs plated with silver. This precaution lessens the liability of debasing the contents of crucibles with iron oxide which may become detached when they are handled with rusty tongues.

We proceed to speak of such lamps as are suitable to laboratory purposes.

Glass, Spirit Lamp.—This is a small glass lamp like the

one shown in Fig. 115. The body is the reservoir for the alcohol. To the neck b is adjusted a copper circular shield c, with a tube in its centre for the passage of the wick, which should be of cotton, and similar to that used for tallow candles. The shield should rest upon, rather than within the neck, otherwise its expansion by the heat may cause the breakage of the glass. A minute opening drilled in the shield is also requisite Fig. 114.

Fig. 115.

drilled in the shield is also requisite for the escape of vapor

#### HEATING OF TUBES BY LAMPS.

in case the alcohol should become heated. The glass cap a, ground interiorly, so as to fit hermetically to the neck of the lamp when not in use, prevents the evaporation of the alcohol and the consequent impregnation of the wick with water, which renders its relighting difficult. The lamp must, however, be invariably extinguished before putting on the cap.



These lamps are useful for heating small apparatus, such as test tubes, Figs. 116, 117, and reduction tubes, Fig. 148, and for larger vessels which require only a gentle heat, as shown in Fig. 118.

Fig. 118.


# BERZELIUS' SPIRIT LAMP.

Berzelius' Lamp. Fig. 26 at page 54 represents this lamp with the improvements recommended by Mitscherlich and Lie-



big. It should be made of thick sheet copper or brass, and brazed instead of being soldered together. Its form is that of an Argand lamp, with a circular body or reservoir g, which receives its fuel through the stoppered opening r. The mechanism contained in the frame work s, and communicating with the cylinder t allows the elevation or depression of the wick at will. The only communication between this portion of the lamp and the reservoir is by a small tube through which the alcohol is supplied to the wick. The chimney u may be movable and adapted to a flattened socket soldered to the side of the inner circumference of the reservoir, or else be hinged in the same position so that it may be thrown back when the lamp is to be lighted or trimmed. Surmounting the chimney is a crucible jacket h with a handle q adapted to the socket or thumb screw. The crucible with its movable cover d, Fig. 120, is

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Fig. 120.

placed in the centre of the sheet iron jacket upon supports so as to receive the full force of the flame. It is designed to protect the crucible from all air save that which passes up the chimney. The whole arrangement is shown by Fig. 120, c being the chimney of the spirit lamp, and the arrows showing the direction of the flame which passes unobstructed upward. All atmospheric air save that which passes up the chimney being excluded, the heating power of the lamp is greatly increased.

> The lamp, as is seen by the figures, is mounted upon a fork v, which slides upon the upright of the support b.

This upright is a smooth wrought iron or brass rod, screw cut at its lower end, and firmly fastened to the walnut foot b by means of a nut. The foot serves as a ballast and at the same time as a bed for a large capsule, which is a convenient receptacle for any matter which may be accidentally spilled from the heating vessel. The pan p is intended for the same purpose when the support is occupied on either side. There are other appliances which add to the convenience of this lamp. They consist of thumb screws and sockets d e f for holding the iron wire rings m l i k. These rings, varying in diameter, serve as supports for the vessels employed, and to steady those which are tall like the flask shown in the figure, a clamp The thumb screw to which the rings are f is requisite. attached slide upon the upright rod and allow the elevation or depression of the heating vessel at will. The iron plate sand bath n is very useful for digestions in beaker glasses which will not safely bear direct contact with the flame.

The fittings of this and all other chemical lamps should combine lightness with strength so as to avoid the dissipation of too much heat by excess of metal.

Fig. 121 exhibits a lamp support not very dissimilar to the preceding, but with a cast iron triangular foot b, of weight sufficient to prevent the lamp from being upset by the superposition of heavy vessels. The iron triangle d is a very convenient substitute for the ring when a crucible is to be heated, as that shape affords a better support. The rod a of brass or iron is from twenty to twenty-four inches in length. The fork g for the lamp, and the rings, are all adapted to the

thumb screws which hold them steadily until they are to be replaced by others of different form or size for different and larger vessels.

A very convenient modification of Berzelius' lamp for boiling in large vessels is shown by Fig. 122. It is supported by three feet of solid brass. Adjusted to its wooden handle is a brass crook for supporting the necks of beaked vessels, retorts, and the like. This crook can be lowered or elevated at will by means of the thumb screw by which it is fastened. Two rings accompany it, one of open work for the support of capsules, broad and round bottomed vessels; and the other cullendered with fine holes for the distribution of the heat to flat bottomed glass vessels. Fig. 121.

This is a powerful lamp, and is more convenient for large vessels than the lamp mounted as before described. Luhmè,



who first recommended this form, also advises that there be no direct communication between the reservoir and the circular space containing the wick, because such an arrangement is promotive of accidents. When the lamp has burned for a length of time and nearly all the alcohol is consumed, the reservoir becomes filled with vaporized spirit, which may explode when it is re-lit after being refilled. All this is prevented by forming the connection by means of a tube. The Berzelius lamps of recent manufacture are made with this improvement.

Either of these lamps, and all others in which spirit is consumed, must be provided with a metallic extinguisher to protect the wick and prevent evaporation of the alcohol. This extinguisher is seen in Fig. 26.

Rose's Lamp.—This lamp, also constructed upon the principle of the Argand burner, gives an intense heat. It pos-

Fig. 123.



sesses the advantage recommended by Luhmè of having the reservoir at a distance from the burner, so that the spirit remains unheated during the longest operations. The wick is regulated by a rack and pinion as in the Berzelius lamps, and its mode of management is precisely the same. Mr. Kent, of New York, who manufactures them, annexes the improvement of Prof. Horsford, by which a heat is produced sufficiently intense for bending glass or fusing carbonate of soda in a few minutes. The copper reservoir is used by being placed upon a tripod accompanying it, and fitting to the Rose lamp as shown

by Fig. 124. Three fluid ounces of alcohol are poured therein, and the screw plug tightly closed. The jet is cleaned by probing it with a needle, and the heat of the lamp is applied until the alcohol boils. The vaporized spirit passes through the jet, and when the chimney is on, takes fire, and produces a blast flame of great power. The contents of a platinum crucible placed about half an inch above the chimney, as shown in the figure, becomes fused in a few minutes.

The Russian Lamp.—This apparatus, Fig. 125, of Russian invention, and similar in principle to Horsford's "Cambridge

blast lamp," is said by Noad to afford a very powerful heat in a few minutes. It consists of a strong double brass cylinder or box, the interior arrangement of which is shown by the dotted lines in the cut. A piece of tube terminating in a jet passes from the exterior to the interior chamber, rising nearly to the top of the former. The fuel is supplied through the aperture b, closed with a cork, and not with a brass cap. The lamp is known to be fully charged when the spirit begins to flow from the jet. The inner chamber is then to be filled with the same spirit to within half an inch of the apex of the jet. The ignited spirit in the inner chamber heats that in the outer, and causes it to boil, and the pressure of the vapor forces the boiling spirit through the jet in a powerful stream, which of course becomes immediately inflamed, and acts as

an energetic blast, producing heat enough to ignite a platinum crucible placed above it to whiteness. The triangle

which supports the crucible must be of platinum, and the ring upon which the triangle rests of very stout iron wire in order to resist the fusing effect of the flame.

There are certain precautions necessary in the use of this lamp, to guard the operator against accidents. Before introducing the alcohol, it is proper to be assured that the jet is free from impediment by blowing through it. The

cork stopper b must be put in rather loosely, so that it may





offer no resistance should a stoppage occur during the operation. For still greater safety, that part of the lamp should be turned from towards the experimenter.

Pyroxylic spirit<sup>\*</sup> is the fuel recommended; and it is said that a lamp of this construction,  $3\frac{1}{2}$  inches in height, and  $3\frac{1}{4}$ in diameter, will burn with a charge of four ounces of spirit for thirty minutes, which is long enough for most fluxions with carbonated alkali.

The Gas Lamp.—This arrangement, Fig. 27, which has been fully described at p. 54, supersedes all other heating apparatus for table operations. Crucibles, capsules, and retorts are alike readily heated by it, and even distillations on a large scale may be successfully performed. To effect the latter object, the upper half of a black lead or clay crucible may be placed around the lamp, provided with an opening on one side for the beak of a retort to pass out. The lower half of the crucible, with its bottom broken off, is then inverted over the whole, and the hole at the top loosely covered to allow of the escape of the products of combustion. By this arrangement, the heat of the flame reverberates through the dome, and increases the effect to such a degree that several pounds of mercury may be distilled at once from the red oxide.

The Table Blow Pipe.—This table, shown in Fig. 30, and described at p. 59, may be used either with a Berzelius or Rose lamp, or the Argand gas burner, Fig. 31. Gas,<sup>†</sup> when

\* Pyroxylic spirit is a very inflammable alcoholic compound, obtained as one of the products of the destructive distillation of wood. As found in commerce it is impure. (See Ure and Turner.)

<sup>+</sup> As the use of alcohol for lamps and of fuel for furnaces, with the necessary attendance upon the latter, involves a considerable expense, annually, even in an experimental laboratory, it is not inappropriate to allude here to an economical means of replacing them with gas.

The material which may be used for this purpose is the refuse fat of the kitchen, a gallon of which in its melted state will yield 100 cubic feet of oil gas, sufficient to feed a bat wing burner for upwards of seventy hours.

Its greater density and amount of olefiant gas give it superiority over the coal and rosin gas. Moreover, it would be difficult to adapt an apparatus to the manufacture of small quantities from the latter materials. Fig. 126 exhibits Kent's portable apparatus for the manufacture of gas from grease. It consists of a wrought iron retort, thirteen inches high and six inches in diameter, with a large opening at the top for the passage of lumps of coke with which it is to be three-fourths filled. The coke is used to increase the extent of the heating surface so as to facilitate and hasten the generation of the gas.

The retort is to be heated to low redness, but no higher, otherwise the gas becomes decarbonized. The oil is supplied to the retort in a thin but constant stream from the funnel, through a small hole in the key of the stop-cock. The

# MANUFACTURE OF ILLUMINATING GAS FROM GREASE. 169

furnished by public companies, is by far the most economical source of heat, and withal is powerful, readily manageable, and cleanly. For all the nicer ignitions, fluxions, and fusions it does away with the necessity of a furnace, which is less convenient, and requires tenfold the time for its action. In five minutes, by the use of this implement, we can often satisfactorily complete processes which with a furnace would require an hour. This saving of time and fatigue is an important consideration when the operations are to be multiplied or rapidly repeated. It is applicable to all the purposes of ignition, fusion, and fluxion of limited quantities of matter, and by "driving the current of air obliquely and somewhat downward through the Argand burner, the process of cupellation may be accurately performed on three hundred grains of lead."

retort is connected by an iron tube with a copper reservoir immersed in cold water, for the purpose of cooling the gas and collecting a portion of undecomposed oil which passes over. An additional receiver renders this apparatus applicable to the purposes of illumination or heating. All the pipes and appliances for either are furnished with the apparatus by the manufacturer, to order. The retort requires to be occasionally cleansed, but at no other time has it to be necessarily opened.

Fig. 126.

Large vulcanized India-rubber bags make excellent gasometers for small quantities of gas, but for 100 cubic feet, it will be better to have a sheet iron bell well payed over, internally and exteriorly, with plumbago paint. The cistern for its reception can be sunk in the yard, and for the above quantity its dimensions must be  $6\frac{1}{2}$  feet diameter and  $4\frac{1}{2}$  feet depth.

# 170 CRUCIBLE HEATED OVER BLOW-PIPE FLAME.

When gas is used it is only necessary to bring the Argand burner, Fig. 31, over the jet 3, Fig. 30, and to depress it



so much that its orifice may extend a short way into the flame for heating a vessel of small surface, and still further for vessels of greater superficies. The gas being turned on and inflamed, the treadle is then worked with the foot, slowly at first, until the current of air thus forced up through the tube changes the white and quiet flame into one of a pale reddish tint and ragged outline. If too much air be driven through, the flame becomes bluish, and the heat becomes less intense.

When a lamp is used, it is necessary that it should have a circular Argand burner, which is to be placed over the jet 3, in such a position that the orifice of the latter projects through the centre of the burner, just beyond the top of the wick. The length of the flame being proportional to the elevation of the wick, the latter must be adjusted accordingly by the screw and rack before being ignited. The flame being of the proper height, the treadle 4, Fig. 30, is to be worked at first slowly, for the heat must be gradually applied, and then more rapidly by increasing the motion of the foot until the blast produces a buzzing sound, when the impulse is continued or moderated as the case may require.

The crucible to be heated is placed upon a wire triangle,

resting upon a ring of an upright support, as shown in the figure, and is placed over the FLAME, so that it may be surrounded by the upper or hotter portion (BLOW-PIPE). If the flame is smoky, and deposits carbon upon the sides of the crucible, the blast must be increased or the flame lowered.

The operation being finished, the covered crucible is left to cool before being opened.

Compound Blow-Pipe.—This apparatus, known as Hare's oxyhydrogen blow-pipe, is used for the fusions of such refractory but fusible substances as resist the highest power of the furnace. Its action is based upon the intense heat produced by the ignition of combined oxygen and hydrogen gases.

Dr. Hare's form of apparatus, with which he fused twentyeight ounces of platinum in one mass, is given and fully treated of in his *Compend*, and also in the *Encyclopedia of Chemistry*; but our remarks will refer to a more economical instrument constructed upon the same principle.

Fig. 128 exhibits the instrument as made and sold by Kent.



It consists of two vulcanized India rubber bags or reservoirs, of twenty gallons or greater capacity. These bags are very flexible, strong, and portable; one of the above size, when empty, occupying but a very limited space. They are filled, the one with oxygen, and the other with hydrogen gas, each being fitted with a connecting screw and stop-cock, by

## GENERATION OF OXYGEN GAS.

which they can be adjusted directly to the generating apparatus, as shown by Fig. 129, or with a gasometer, when they



are to be charged. The communication between the bags and the jet-pipe above the table is by means of the flexible India rubber or lead tubes, coupled by gallows screws, Fig. above. The jets are so divided within the pipe that the gases enter at opposite ends, and consequently are not mixed until they meet at the arm of the jet, which is so arranged that it can be raised or lowered on an ordinary retort stand. By this arrangement, which is a convenient modification of the old double jet, a jet of oxygen passes through the centre of a circular flame of hydrogen, the mixture and consequent explosion of gases being avoided.

The gradual efflux of the gas from the reservoirs is effected by superposed weights—a much more convenient mode than that of hydrostatic pressure, which is requisite when metallic reservoirs are used.

The two gases are very readily prepared. The whole apparatus for oxygen is shown in Fig. 129. The retort, a thin copper flask, is connected with a brass cap and neck by a gallows screw.

Four ounces of good chlorate of potassa, and one ounce of peroxide of manganese are mixed together and placed in the retort, the cap screwed down, and the joints luted with pipe clay. The heat of a Berzelius lamp, applied as shown in the figure, drives over ten gallons of pure oxygen in fifteen minutes.

The lamp may be removed as soon as the gas begins to be

#### GENERATION OF HYDROGEN GAS.

generated and pass over freely, as sufficient heat will be retained for the completion of the operation. The *caput mortuum* of chloride of potassium and oxide of manganese remaining in the retort can readily be removed by water.

Hydrogen can still more readily be obtained from a selfregulating reservoir. Fig. 130 exhibits the apparatus as made



by Kent. The copper cylinder which he uses is replaced in other similar instruments by glass. It consists of a japanned copper cylinder, 9 inches high, and 6 inches diameter, with a cover and bell attached.

Within the bell hangs a basket of copper wire, which is to be filled with about  $\frac{3}{4}$  lb. of zinc, in lumps. The outer cylinder is to contain 6 lbs. of cold diluted sulphuric acid, made with 1 part acid to 4 of water.

In the upper part of the bell is a division, forming a receptacle for a strong solution of potash, 2 f.3 of which are put into it through the opening in the top, which is then to be tightly stopped.

The apparatus being adjusted, and the stop-cocks opened, the dilute acid rises in contact with the zinc, and generates hydrogen gas, which, being forced through the potassa solutions, becomes washed previous to its exit from the stop-cock into the bag. An hour suffices to generate twenty gallons of gas; and, when it is desired to arrest the operation, close the stop-cock so as to produce an accumulation of gas in the bell, and thus displace the acid. The action may be renewed at any time by opening the cock.

Accompanying this apparatus, as shown by separate figures in the cut, are two convenient addenda for other purposes; one a jet, with platina sponge and fixtures, for producing an instantaneous light, and the other for bending glass tubes, &c. They are both readily attached to the stop-cock by their screws. In the first case, the gas is projected against the platina sponge, and becoming immediately ignited, affords a ready means of lighting a taper. The sponge, when not in use, should be kept covered and dry.

The oxyhydrogen blow-pipe is put into operation by first charging the bags with gases, placing on their weights, letting on the hydrogen, igniting it, and passing a jet of oxygen through the flame directed upon the substance under process. This substance should rest upon charcoal or fire-brick, and in a cavity drilled for the purpose so as to prevent its being blown away by the force of the blast. For the same reason, when the substance is in powder, it is necessary to moisten it with water, and compress it in the cavity. The charcoal rest is very conveniently supported upon one of the sliding rings (Fig. 131), which allows the facility of bringing it near to the orifice of the pipe where the combustion takes place.

When this apparatus is used for the purposes of illumination, as in the production of the *Drummond light*, which is produced by the action of its flame upon a cylinder of lime, the nozzle of the blow-pipe must be pointed upwards, so that the flame may have full play upon the incandescent earth.



Supports.—In lamp, table furnace, and blow-pipe operations, vessels are maintained over the fire by supports, differing in material and construction with the uses for which they are destined.

Å very simple and economical support is shown in Figs. 131 and 132, the only difference between the two being in the shape of the foot, one being rectangular and the other round. It consists of an upright iron rod, from 20 to 24 inches long, and about  $\frac{1}{3}$  of an inch or more in diameter, screwed into a cast iron foot, and fastened beneath by a nut. The three projecting rings, of iron wire, 2, 3, and 4 inches in diameter, are held by thumb screws, which permit their elevation or depression at will.

For the larger stands the thumb screw is of iron, and of the form exhibited in Fig. 127 and Figs. 133, 135, b. It is made Fig. 133.



with two holes, at right angles to each other, and screws, one for the reception of the iron upright, and the other for the handle of the ring, which can readily be detached and replaced by another of different size. This form of screw and socket prevents the necessity and expense of having the rings attached to the screws. One of these screws will answer for a series of rings, and the latter being of iron wire, the operator can readily form them himself of any required shape. With

this arrangement, and a series of different sized rings, the support is convenient for all its purposes without the expense of a socket for each ring. When the rings are too large for the vessels to be heated, their diameters may be diminished by means of stiff wire triangles, Fig. 134. They are particularly useful for the support of small crucibles, as is shown in Fig.





27, p. 54. There should be a number of them of different sizes.

Fig. 135 exhibits what is called the "universal support." The foot is of cast iron, and the toes twelve inches apart. The upright rod is of iron, 36 inches long, and 3 in diameter. It is substantially made for the support of heavy capsules, retorts, &c. The thumb screw and socket b are of solid brass or iron. A brass vice. 6 inches long, and  $1\frac{1}{2}$  inches wide, lined in its mouth with buckskin, is shown at g. It is fitted by the arm f to the hole c, and serves for the support

of heavy retorts and other beaked vessels, as shown in the figure. Three solid brass rings, of from 4 to 7 inches diameter, accompany this stand, and are held in the socket d by the thumb screw a. As the arm of each of these rings is adapted to the socket, one may replace the other when it is desired to change the size.

Wooden supports adapted for tube arrangements, made of box wood or hickory and lined with cork or buckskin at the parts intended for grasping, are also convenient and necessary pieces of apparatus. They consist of foot rod and nut similar to the iron supports. Their other parts are a wooden vice (Gay-Lussac's), Fig. 138, for supporting the necks of re-



torts and other beaked vessels; Gahn's cylinder holder, Fig. 137, for experiments with gases; and a wooden ring, Fig. 136, as a rest for inverted flasks. Fig. 139 exhibits an upright retort clamp with a movable joint and wooden screw by which it can be raised or lowered at pleasure. The stand or lower portion is similar in construction to D E, Fig. 142.

Fig. 140 represents another support with two sliding arms, the upper one of which is a filtering stand, and the lower a tube or retort holder. The funnel supports of the upper arm are adjusted thereto by means of wooden thumb screws, and may be removed at pleasure. These are less convenient than the single filter stand, Fig. 141, though they have the advan-



tage of allowing several simultaneous filtrations upon one arm, and thus a single stand may do the service of three or four according to the length of the arm. The uprights of these supports should be screw cut at the lower end and fastened to their pedestals by means of nuts, which retain them in place more firmly than any other mode.

Another form of stand is that known as Berzelius' table support. It is of hard wood and consists of a loaded foot D, Fig. 142, supporting a flat disc A, which by means of its leg and the thumb screw E can be raised or lowered to any desired height. This is a very convenient rest for a small lamp or furnace or for recipients in distillations when it is required to raise either above the level of the operating table. When in this or other cases the surface of the supported vessel is round, it

#### TUBE AND BULB RESTS.

Fig. 142.



Fig. 143.



Fig. 144.



is safer to steady it upon a braided straw ring, Fig. 143, interposed between its bottom and the disc.

For supporting tubes and other vessels horizontally, the disc may be replaced by the brass crook as shown in figure 144; and for globular vessels and flasks by the wooden tripod, Fig. 145. Each of these pieces is adapted to the stand D, and one may replace the other when necessary, the screw allowing them to be raised and steadily maintained at the required height. The height of this instrument when drawn out to its full length is twenty inches.

As a support for large evaporating vessels over furnaces, an iron tripod, Fig. 146, is very convenient.

The test tube stand or rack, which is the only support that remains to be mentioned, has been alluded to before, p. 54,



Fig. 25. A smaller one for table use is shown in Fig. 147. It consists of two narrow uprights, say ten inches high, of thin poplar wood, which are braced together by three shelves. These shelves are perforated throughout their length with auger holes for the reception of the test tubes. The smaller and shorter tubes occupy the upper range, the interval between which and the middle shelf should be less by an inch than that between it and the lower.

The manifold uses of the preceding instruments will be further explained when treating of manipulations to which they are applicable. At present a familiar idea may be obtained by reference to Figs. 116, 118, 119, and to the



cut below in which several of them, as applied in operations, are included.



# CHAPTER XII.

#### BATHS.

THE preceding chapter relates to apparatus for the direct application of fire; our present remarks will refer to the means by which the heat is moderated and diffused before it is allowed to reach the substances under process. These means consist of baths, which are called vapor, water, saline, metallic, oil or sand, according to the nature of the interposed bodies employed.

The object of an intermedium is to prevent a too rapid application of heat, and to give greater uniformity of temperature. Inequality and too sudden application of heat being thus provided against, the fracture of vessels and ejection of their contents, and many inconveniences attending other modes of applying heat are avoided.

Baths are very convenient for heating substances which require a constant and somewhat permanent elevation of temperature, and which might undergo decomposition over the naked fire. The temperature to be obtained, is, however, only limited, but by a proper management of the fire any degree of heat up to the maximum amount which the intermedium is capable of receiving from the means employed, can be obtained.

Baths consist of two vessels of different diameters, and they may be either of metal, stoneware, or porcelain. The outer jacket receives the intermediary body, and the inner one the substance to be heated. As generally constructed, baths are made with but one jacket, the aperture or mouth being of diameter corresponding with that of the heating vessel which is to be placed over it. This arrangement obviates the necessity of an inner jacket and also of the transfer of the substance which is being heated.

Baths are useful in operating upon organic and other bodies easily alterable by heat. They are also convenient for determining the melting point of those substances which are fusible at or below the degree of heat, which can be imparted to the liquid or vapor of the bath: that temperature being made known by immersing a thermometer at the commencement of the fusion and noting the degree.

In the fluid baths the thermometer may be permanently fixed by means of a perforated cork, closing a circular aperture in the lid. Fig. 84 with the scale upon the stem exhibits the most suitable form of the instrument for this purpose. The difference in temperature between the bath and the heating substance, which is sometimes very considerable, especially when porcelain or glass is used, may be diminished by keeping the bath always covered. If the vessels have smooth surfaces, the heat obtained previous to ebullition is much higher than in the opposite case. While the bath is in use, care must be taken that the amount of the liquid in the outer vessel remains as nearly as possible the same throughout the process, and the loss from evaporation or exit of vapor should be replaced by frequent but gradual additions of the same fluid.

Steam-bath.—This apparatus forming a fixture of the laboratory has been fully described at p. 42, Fig. 11. It is a convenient arrangement for heating those substances which would be alike injured by the direct application of fire or contact of steam. It affords a temperature of fully 212° F.

For very small operations, the apparatus of Dr. Ure, Fig. 149,

is an excellent contrivance. "It consists of a tin box about 18 inches long by 12 broad and 6 deep. The bottom is hollowed a little by the hammer towards its centre, in which a round hole is cut of five or six inches in diameter. Into this a tin tube three or four inches long is soldered. This tube is made to fit tightly

into the mouth of a common tea-kettle, which has a movable handle. The top of the box has a number of circular holes cut in it of different diameters, into which evaporating capsules of platinum, glass, or porcelain are placed. When the kettle filled with water and with its nozzle corked is set on a stove, the vapor playing on the bottom of the capsules heats them to any required temperature; and being itself continually condensed, it runs back into the kettle to be raised again in ceaseless cohobation. With a shade above to screen the vapor chest from soot, the kettle may be placed over a common fire. The orifices not in use are closed with tin lids. In drying precipitates, the tube of a glass funnel may be corked and placed with its filter directly into the opening of a proper For drying red cabbage, violet petals, &c., a tin tray size. is provided, which fits close to the top of the box within the rim which goes about it. The round orifices are left open when this tray is applied."

Water-bath. — The water-bath is used for heating those substances which require a temperature lower than that of

Fig. 149.

boiling water. It is very available where bodies easily decomposable by high temperatures are to be heated, and is also useful for the gradual exhaustion of vegetable and other substances which only give up their soluble matter after long contact with the warm solvent liquid. Though seldom employed for the purpose of reducing the temperature very low, it may, by proper management, be made to yield any intermediate temperature from  $32^{\circ}$  to  $212^{\circ}$  F.

Every water and liquid bath, whatever its form and the purpose to which it is to be applied, should be so constructed that the vaporized portions can, if necessary, be confined within the vessel and prevented from escaping by other outlets than the safety valve. As proximity of the escaping vapor might be injurious to the substance under process, the escape pipe should have its exit six or eight inches distant from the sides of the vessel.

Any two vessels of different diameters, one within the other, may form a water-bath, provided the intervening space at the bottom and sides is sufficient to contain the requisite amount of water. Straw at the bottom and sides is an excellent means of steadying the inner vessel and preventing direct contact of its surface with the more highly heated bottom of the outer vessel, a result which would cause the temperature of the inner vessel to be raised above that of the surrounding water.



A regularly constructed water-bath of convenient form is shown in Fig. 150. "A is the vessel containing the water, a the inner flange for the support of the evaporating dish B,

or different circular rings when smaller dishes are employed. d is a tube furnished with a stop-cock for the escape of the steam, which, in some cases, requires to be carried still farther off by an additional tube attached to it. The whole apparatus may be heated either by a gas-stove or on a charcoal furnace. The water-bath is filled about half-full with water. It will be seen that water-baths with cover act more on the principle of steam-baths as soon as the quantity of water which they contain is small. Care must be taken always to replenish the evaporating water by adding fresh, otherwise not only the experiment may be ruined, but the bath itself become seriously injured."

Large tin cups make excellent water-baths for flasks and tall vessels. The bottom of the flasks may rest upon small straw rings which prevent contact with the heated tin and serve also to steady them. The holes in the lid for the protrusion of the necks of the flasks also assist Fig. 151.

in this latter respect.

A very convenient little bath for very small operations, is shown in Fig. 151. It consists of a copper capsule a, with a ledge around its interior circumference as a rest or support for the vessel to be heated. In order that it may be applicable to capsules of any size, its top is

fitted with a series of thick flat copper rings, which afford the power of decreasing the diameter of the outer capsule to suit that of the vessel to be heated.

The whole diameter of the outer vessel is about 7 inches, and when all the rings are placed upon the top the opening in the centre is about 1 inch, but by withdrawing one at a time the size of the mouth can be increased gradually from 1 to  $6\frac{1}{2}$  inches and adapted to any vessel of intermediate size. It would be well to have a small tube projecting from the side so as to answer at the same time the purpose of a handle, and of an exit pipe for the waste vapor.

This apparatus, as is seen in the figure, is mounted upon a tripod b, a convenient support when the small spirit lamp is used to heat the water. When a stronger heat is required it can be detached and mounted upon a larger support and placed over the gas or Berzelius lamp. Fig. 152 represents the apparatus without the tripod and with handles.

A porcelain or tin plate placed upon the top of this bath



## SALINE BATHS.

renders it very convenient for drying small filters, precipi-

Fig. 152.



tates, &c. By increasing the circumference of the bath so that it may have a broad top, and by piercing the latter with circular holes of various diameters, we obtain a means of heating several capsules of different sizes at the same time. Water-baths are useful for exhausting organic and other matters readily

destructible by heat. For the completion of evaporations which have been carried to the safest extent upon a sand-bath, they are also very available.

The still, Fig. 13, without its head makes an excellent waterbath for large operations.

Saline baths.—The substitution of saline solutions for water affords a means of obtaining temperatures higher than the boiling point of that liquid. This kind of bath is very useful for digestions and many other operations. The saturation of the water with salts raises its point of ebullition, but in practice it is necessary to use weaker solutions, otherwise the evaporation of the water would be continually causing the deposition of the salt and the solidification of the liquid to the great inconvenience of the experimenter.

The following table exhibits the boiling points of the saturated solution of the salts most generally employed for this purpose.

Alum	-		2200	Carbonate of potassa	-	-	2849
Muriate of ammonia	-		236	Chloride of zinc -		-	320
Oxalate of ammonia	-	-	218	Rochelle salt	-		246
Tartrate of ammonia			230	Sulphate of nickel -		•	235
Chloride of barium			222	Chlorate of potass -	-	•	218
Nitrate of baryta -			214	Nitrate of potass -	-	•	238
Acetate of copper -		-	214	Quadroxalate of potass	•		220
Sulphate of copper -			216	Acetate of soda -	-	-	256
Acetate of lead -	•		212	Nitrate of soda -	-		246
Chloride of calcium	•	•	220	Biborate of soda -		•	222
Sulphate of magnesia		-	222	Carbonate of soda -		- 1	220
Cream of tartar -			214	Phosphate of soda -	•	•	222
Chloride of sodium	-		224	Nitrate of strontia -	-	•	224
Tartrate of potassa	-		224	Sulphite of zinc -	•	•	220
Sulphate of soda -		-	213	Boracic acid	-		218
-							

## BOILING POINTS OF SATURATED SOLUTIONS.

Chloride of zinc is included in the above table, because of is great deliquescence and availability at temperatures below 320° F. Beyond that degree it gives off fumes of chlorhydric acid and becomes inconvenient as a medium for the communication of heat. A layer of oil retards the evaporation of the water and promotes the elevation of the temperature of the solution.

The selection of the salt for the bath must be with regard to economy and the nature of the material of the vessels. Corrosive solutions should only be used in those vessels upon which they are without action. The construction of the bath is similar to that shown in Fig. 150.

Metallic baths.—These baths are only convenient for small operations, because of the difficulty of supporting the weight of a large amount of metal and of keeping the heating vessels immersed in it. They furnish temperatures higher than either of the preceding, and for greater safety must be heated in cast iron vessels of form suitable to the experiment.

Mercury would be a convenient menstruum, if it was lighter and emitted, when highly heated, less noxious fumes. On these accounts it is but rarely used, and only in test tubes for heating still smaller tubes. The fusible alloy, composed of eight parts of bismuth, five of lead, and three of tin, forms an excellent metallic bath. It melts below 212° F., and affords very high temperatures, for though it oxidizes upon the surface as its temperature is increased, it will bear a white heat without emitting vapors. Tin melting at 441° F. and lead at 609° F., are both available for a temperature above their fusing points.

Oil baths.—Oils in ebullition throw off a very disagreeable vapor, but are otherwise convenient for furnishing temperatures above their boiling point. Care should be taken not to apply heat sufficient for their decomposition. Even at low temperatures they have the advantage over water, of losing less by evaporation and of affording facility in regulating the temperature by means of an immersed thermometer. The oil should, before its transfer to the bath, be heated in an iron capsule for several hours, to expel moisture. The bath consists of a porcelain lined or metallic jacket, of construction exactly similar to that of the water-bath. It affords a temperature of  $570^{\circ}$  F., and the facility of determining its temperature, for which purpose a THERMOMETER is a necessary accompaniment.

Sand-bath.—This bath is of more general application for laboratory purposes than either of the others. Its different forms and their appropriate uses have already been fully de-

### THE PORTABLE LABORATORY.

scribed at pp. 38, 39, Figs. 8, 28, 92, 94. Sand is used, because it is a better resistant of sudden changes of temperature, and affords a uniform heat greater or less as may be required than that of the water baths. Magnesia and ashes are sometimes substituted, but they are too apt to be driven about by the least current of air; the former is only used as a bed for platinum, or porcelain crucibles, which are to be enclosed in Hessian crucibles and ignited.

A sand-bath may be readily constructed by filling an iron pot with sand and placing it upon the charcoal furnace, Fig. 87, or upon the top of the stove which heats the apartment. It is then ready to receive glass or porcelain vessels in which the processes of evaporation, digestion, or distillation may be carried on.

## THE PORTABLE LABORATORY.

Having referred to each separate implement employed as a means of applying heat in the various chemical operations, we now call attention to a convenient, compact, and really elegant apparatus, of German invention and construction, which may very appropriately be styled a portable laboratory. It combines within a less space than four square feet, every requisite for convenient manipulations in any process for which a furnace is required.

We are indebted for the drawing Fig. 153, to Allohr's Commentar ar Preusischen Pharmacopa. The furnace and grate are of iron, and can be set wherever it is convenient, adjoining a flue. The accompaniments are a tin lined copper still with pewter head and worm, a block tin digesting cup; two other digesting cups, one of glass and the other of porcelain, protected exteriorly by copper jackets. A large tinned copper evaporating basin, another similar basin with a porcelain interior, and a half score of other convenient and useful vessels. The furnace is so constructed that the furniture may be heated either by the naked fire or by steam, a generator for which surrounds the body of the still. The cooler for the worm, is a large copper cylinder of some twenty gallons capacity, tinned and arranged interiorly and fitted with pipes, adapting it to all the purposes of maceration, decoction, solu-

#### THE PORTABLE LABORATORY.



Fig. 153.



tion, and ebullition. A sand-bath for heating glass and porcelain vessels also forms part of the apparatus.

Weiss and Schively, No. 43 north Front st. Philadelphia, have lately imported an apparatus somewhat after the plan of our drawing, the first one we believe that has appeared or been offered for sale in this country. We cannot too highly commend these implements to the attention of the druggist.

# 188 THE MODE OF PRODUCING LOW TEMPERATURES.

Where the means will justify the outlay, every pharmaceutical workshop should be provided with one. It would not only facilitate the preparation of products, but would also afford the means of pursuing investigations, and thus of inducing the operator to contribute to the advancement of science.

# CHAPTER XIII.

## THE MODE OF PRODUCING LOW TEMPERATURES.

A LOW degree of temperature is often as necessary in some chemical processes as an elevated one is in others. The means of obtaining it are by freezing mixtures, and these mixtures are formed of materials prone to liquefaction. The abstraction of the heat requisite for this purpose, from the bodies with which they are in contact, produces in them a corresponding decrease of temperature.

In many chemical investigations they are particularly useful, especially for determining the freezing point of substances, and as cooling media for the recipients in the various processes of DISTILLATION. The components of freezing mixtures should be finely pulverized, and the whole formed as rapidly as possible. Bulbs and small vessels with their contents may be cooled, but not economically, by keeping their surfaces moistened with ether or some other very volatile matter which, by rapid vaporization, can carry off a large amount of heat.

The following convenient tables by Walker and Karsten, exhibit the composition of numerous freezing mixtures and the degree of cold which they produce.

	Mixtures.		Thermometer	sinks.	Degree of cold produced.
5	Snow or pounded ice - 2 pa Muriate of soda 1	irts {	•••	to —5°	*
5	Snow or pounded ice - 5 4 Muriate of soda 2 4 Muriate of ammonia - 1 4		- - nperatur	to —12°	
Į	Snow or pounded ice 24 " Muriate of soda - 10 " Muriate of ammonia - 5 "	; }	iny ten	100	
	Nitrate of potash 5 " Snow or pounded ice 12 "	• ]	From 2	to —18°	
3	Muriate of soda - 5 4 Nitrate of ammonia - 5 4	: }	••••[	to -25°	•
52	Snow 3 " Diluted sulphuric acid* - 2 "	}	From +32°	to —23°	55°
52	Muriatic acid (concentrated) 5	5	From +32°	to —27°	59
32 6	Concentrated nitrous acid 4 "	5	From +32°	to -30°	62
300	Muriate of lime - 5 " Snow 2 "	5	From + 32°	to40°	72
25	Crystallized muriate of lime 3 " Snow 3 "	• • •	From +32°	to -51°	83
1	Potash • • • • 4 "				

TABLE I. Consisting of Frigorific Mixtures, composed of Ice, with Chemical Salts and Acids.

N.B. The reason for the omissions in the last column of this table is, the thermometer sinking in these mixtures to the degree mentioned in the preceding column, and never lower, whatever may be the temperature of the materials at mixing.

**TABLE II.** Consisting of Frigorific Mixtures, having the power of generating or creating Cold, without the aid of Ice, sufficient for all useful and philosophical purposes, in any part of the world at any season.

	Mixtures.			Thermometer sinks.	produced.
	Muriate of ammonia Nitrate of potash Water	- 5 - 5 16	parts "	From $+50^{\circ}$ to $+10^{\circ}$	40°
	Muriate of ammonia Nitrate of potash Sulphate of soda Water	- 5 - 5 - 8 16	22 22 24 24 24 24	From $+50^{\circ}$ to $+4^{\circ}$	46
3	Nitrate of ammonia Water	- 1 - 1	66 66	From $+50^{\circ}$ to $+4^{\circ}$	46
( < (	Nitrate of ammonia Carbonate of soda - Water	- 1 - 1 - 1	66 66 65	From $+50^{\circ}$ to $-7^{\circ}$	57

\* Strong acid 2 parts; water or snow 1 part, by weight.

## FREEZING MIXTURES.

	Mixtures.		Thermometer sinks.	Degree of cold produced.
Se	Sulphate of soda - Diluted nitrous acid*	• 3 parts }	From $+50^{\circ}$ to $-3^{\circ}$	530
{	Sulphate of soda - Muriate of ammonia Nitrate of potash - Diluted nitrous acid	- 6 " - 4 " - 2 " - 4 "	From +50° to -10°	60
5	Sulphate of soda - Nitrate of ammonia Diluted nitrous acid	- 6 " - 5 " - 4 "	From +50° to -14°	64
S	Phosphate of soda - Diluted nitrous acid	• 9 " • 4 " }	From + 50° to -12°	62
5	Phosphate of soda - Nitrate of ammonia Diluted nitrous acid	-9 " -6 " -4 "	From +50° to -21°	71
ş	Sulphate of soda - Muriatic acid	- 8 "	From +50° to 0°	50
Se	Sulphate of soda • Diluted sulphuric acid†	• 5 " • 4 "	From $+50^{\circ}$ to $+3^{\circ}$	, 47

N. B. If the materials are mixed at a warmer temperature than that expressed in the table, the effect will be proportionately greater; thus, if the most powerful of these mixtures be made when the air is  $+85^\circ$ , it will sink the thermometer to+2°.

TABLE III. Consisting of Frigorific Mixtures selected from the foregoing tables' and combined so as to increase or extend Cold to the extremest degrees.

Mixtures.	Thermometer sinks.	Degree of cold produced.
Phosphate of soda - 5 parts Nitrate of ammonia - 3 " Diluted nitrous acid - 4 "	From 0° to -34°	34°
Phosphate of soda 3 " Nitrate of ammonia - 2 " Diluted mixed acids - 4 "	From	16
Snow 3 " Diluted nitrous acid - 2 "	From 0° to -46°	46
Snow 8 " Diluted sulphuric acid - 3 " Diluted nitrous acid - 3 "	From $-10^{\circ}$ to $-56^{\circ}$	46
Snow 1 " Diluted sulphuric acid - 1 "	From -20° to -60°	40
Snow 3 " Muriate of lime 4 "	From +20° to -48°	68
Snow 3 " Muriate of lime 4 "	From +10° to -54°	64
Snow 2 " Muriate of lime 3 "	From -15° to -68°	53

\* Fuming nitrous acid 2 parts; water 1 part, by weight. † Equal weights of strong acid and water.

## FREEZING MIXTURES.

Mixtures.	Thermometer sinks.	Degree of cold produced.
Snow 1 part Crystallized muriate of lime 2 "	From 0° to -66°	66 <b>°</b>
Snow - 1 " Crystallized muriate of lime 3 "	From -40° to -73°	33
Snow 8 " Diluted sulphuric acid 10 "	} From -68° to -91°	23

*Remarks.* The above artificial processes for the production of cold are more effective when the ingredients are first cooled by immersion in other freezing mixtures. In this way Mr. Walker succeeded in producing a cold equal to 100° below the zero of Fahrenheit, or 132° below the freezing point of water.

The materials in the first column are to be cooled, previously to mixing, to the temperature required, by mixtures taken from either of the preceding tables.

The following table by Karsten, shows the diminution of temperature in degrees Fah., where 1 pt. of salt is dissolved in 4 pts. water :---

#### FREEZING MIXTURES.

Salts.								Degrees of cold.
Nitrate of	lead -			-	-		-	· 3·4°
66	baryta -	-			-	-	-	- 3·8°
Common s	salt -	-	-	-		-	-	- 3.80
Sulphate o	f copper	-	-	-	-	-	-	- 4·0°
τι.	potassa	-	L	-	-	-	-	- 5·2°
66	zinc -	-		-		•	-	- 5·6°
66	magnesia	-	-		-		-	- 8·1°
Muriate of	baryta	-	-	-	-	-	-	- 8·1°
Sulphate o	f soda -	-	-	-		-	-	- '14·6°
Nitrate of	soda -	-		-	-	-	-	- 17·0°
"	potassa	•		-	-	-	-	- 19·1°
Chloride o	f potassium	-		-	-	-	-	- 21·3°
Nitrate of	ammonia		-	4	-	-	-	- 25·4°
Muriate of	ammonia	-	-	-	-	-	-	- 27·3°

Salts.			Sat. solution of		De	egrees of cold.
Sal ammoniac	•	-	Common salt -	-	1.	15.10
66		-	Saltpetre		-	22.70
Saltpetre -	-		Sal ammoniac -			17.50
	-	-	Common salt -	4	-	16.90
" -			Nitrate of soda -		-	12.70
	-	-	" barvta	-	-	17.50
/ " -		-	" lead -		-	17.10
Glauber's salt	-	-	Common salt -	-	-	8.50
Common salt	-		Blue vitriol -		-	7.40
Nitrate of soda			Sal ammoniac -		-	16.40
66	-		Saltpetre		-	16.60
46		-	Common salt -			14.00
66	-		Muriate of baryta			4.90
66	-		Nitrate of lead -			14.40
Nitrate of barvta		-	Saltpetre			1.350
Sulphate of zinc			Sulphate of potassa			3.10

The following table, by Karsten, of 1 pt. salt in 4 pts. of a saturated solution, shows an increase of temperature :---

Salts.			Sat. solution of	Deg	Degrees of heat.			
Common	salt	-		Sal ammoniac			-	8.20
٤٤		-	-	Glauber's salt				3.10
**	•	-	-	Saltpetre -	•	-	-	1·35°
66			- 1	Nitrate of soda		ī.,		6·8°
Muriate o	of baryta		-	"		-	-	1.150

By mingling solid lead amalgam with solid bismuth amalgam, whereby they become liquid, Orioli obtained  $39.6^{\circ}$  of cold. Döbereiner mixed 204 pts. lead amalgam (103 lead, + 101 mercury) with 172 pts. bismuth amalgam (71 bismuth + 101 mercury), and obtained a diminution of from 68° to 30.2; and by adding to the same 202 pts. more of mercury, the temperature fell to 17.6°. By dissolving the powders of 59 pts. tin, 103.5 pts. lead and 182 pts. bismuth, in 808 pts. mercury, the thermometer falls from 63.5° to 14°.

# CHAPTER XIV.

### FUSION.

THE liquefaction of bodies by heat, a preliminary step to many processes, is termed fusion. *Igneous* fusion applies to the melting of anhydrous substances, and *aqueous* fusion to the liquefaction of a salt in its water of crystallization.

The modes of performing the process, and the material and form of the apparatus employed, vary with the nature of the substance to be acted upon. The chief point to be attended to is the selection of such containing vessels as are not injuriously affected by the fused substance—and which do not themselves react upon their contents.

The implements for fusion are called *crucibles*, the smaller of which, for the less refractory substances, may be heated over the GAS or SPIRIT LAMP. To effect the liquefaction of bodies difficultly fusible or of large quantities of matter, a FURNACE is requisite.

The size of the crucible should be proportional to the quantity of matter to be heated in it. It is best that its capacity should be no greater than sufficient for the contained substance with enough margin to allow for swelling or foaming.

CRUCIBLES.—A crucible, to be available for any and every operation should possess the quality of compactness in order

# CLAY—HESSIAN—LONDON—FRENCH CRUCIBLES. 193

to resist the corrosive action of fused substances, the permeability of gases and liquids, the fusing power of intense heat, and the tendency to fracture by sudden changes of temperature.

It is impossible to combine all these requisites in any one kind of crucible.

The materials of which crucibles are formed are either *pure* clay, or clay mixed with charcoal, quartz, graphite or coke, to render it more refractory. Black lead, porcelain, silver or platinum, have each and all their appropriate application.

Clay Crucibles.—The Hessian and French crucibles are those of this description, which are most used. The Hessian, so called from the place of their manufacture in

Germany, are either in the form of a tapering cylinder or triangular, and are of that kind of crucible most commonly found at our drug shops. They are met with in nests of a half dozen or more, gradually increasing in size from the smallest (of an ounce) to the largest, of pint or quart capacity.

They are grayish yellow or whitish, rough to the touch, and should give a clear ring when held by the bottom and sounded on the sides. Being hard and impermeable, they are very useful for rough fusions; but the silica which they contain renders them unfit for metallic oxides, with which at high heat it combines.

The Hessian crucibles require careful usage, as they are liable to be fractured by even slight changes of temperature. Therefore, notwithstanding their great cheapness, the London or French crucibles are more preferable for nice operations.

The London crucibles are very refractory, regularly formed with smooth surfaces, and will endure a very high heat.

The French crucibles, Fig. 155, which are manufactured by Beaufaye, of Paris, are said to be far superior

to either of the preceding. They are whitish, well-shaped and smooth throughout, and being nearly free from oxide of iron, and less rich in silica, are applicable for the fusion of nearly all substances except certain salts, which, owing to the porosity of the crucible material, are readily absorbed.

Being capable of supporting extreme heat as well as sudden changes of temperature, they are very Fig. 155.



Fig. 154.

useful for the reduction of oxides and fusion of metals. Borax, glass and similar substances remain perfectly colorless when melted in these crucibles.

The mixture of graphite or coke with the clay, which is found in those of Austin's make, renders them capable of better supporting the softening influence of the wind furnace and withstanding the most sudden changes of temperature, but the proportion of the latter must not exceed 33 per cent., otherwise its combustion by the fire will leave the crucible porous and fragile.

As metallic oxides are reducible when hot, by contact with carbonaceous matter, these crucibles, when used for heating those substances, should be lined with a thick coat of clay paste and dried.

Charcoal is the only proper fuel for earthen crucibles, as coke is apt to form scoriæ which attach to the crucible and impede the draught.

Black Lead Crucibles. Blue Pots.—Black lead or plumbago when mixed with one-fourth of its weight of refractory clay becomes capable of supporting intense heat and sudden changes of temperature. The chief use of crucibles made of this substance is in metallurgy, for the purposes of which their smooth surface admirably adapts them. They are not sufficiently compact for the fusion of salts.

Porcelain Crucibles.—Crucibles of this material are very neat implements, but by reason of their incapability of resisting even slight changes of temperature, are only used for purposes to which those of more refractory material are for other reasons not adapted. For heating over the lamp they must be small and thin. In analytic and nice operations they replace platinum in many processes in which the contents act upon that metal, for example, in igniting plumbic precipitates, melting metallic oxides with sulphobases, preparing enamels, and heating metallic oxides which are reduced easily in contact with platinum.

The crucibles, Figs. 156, 157, used directly over the lamp,



should never exceed an ounce in capacity, for even with the most careful management it will be difficult to cool one of larger size gradually enough to prevent its breaking. Berzelius recommends their insertion in platinum crucibles as a means of diminishing their

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fragility. The French porcelain being very thin and light, and a better supporter of sudden changes of heat, is preferable to the Berlin for small crucibles.

The impermeability and cleanliness of these crucibles, render them very convenient for the fusion of certain nice substances, such as nitrate of silver, potassa, &c., in large quantities, and as it is impracticable to have a very large platinum crucible in private laboratories, one of porcelain is substituted. These large crucibles, made with covers, may be of either of the forms, Figs. 158, 159, 160, and of Berlin porcelain, which is similar to wedgewood-ware, and heavier and cheaper than the French.



The large crucibles, varying in size from two to six inches in height, and from one to four inches in diameter, may be entirely biscuit or else glazed only internally, and if heated over the fire require to be enclosed in a refractory fire-clay case, as shown in Fig. 161. This case is equally useful for platinum or silver crucibles (Fig. 120), as it gives them a proper elevation above the grate and prevents contact with the coals.

For the heating of more readily fusible substances they may be imbedded in a sand-bath and heated up gradually. If allowed to remain in the bath until it has cooled the liability of fracture from sudden refrigeration will be diminished.

Some of the crucibles have duplicate covers, one of which is perforated and used to facilitate the escape of the gaseous matter generated during certain processes.

Metallic Crucibles.—Cast and plate iron, silver and platinum are all used as materials for crucibles.

Iron Crucibles.—For the fusion of silicates and certain seleniurets, sulphurets and other substances, iron crucibles are very convenient. An exterior coating of clay is requisite to protect them from the oxidizing action of the air, to

## IRON-SILVER-PLATINUM CRUCIBLES.

which they are subjected at high temperatures. The same object may be effected by inserting them in clay crucibles.



When, however, the heating is not of long duration nor intense they may be used naked.

Those of wrought iron, Fig. 162, are struck up from a single piece of thick sheet metal.

Cast iron crucibles, Fig. 163, are cheaper, and equally as good as wrought iron for medium temperatures, but they must be turned smooth interiorly.

As some of the constituents of stove coal exert a chemical action upon metal, the only proper fuel is charcoal.

Silver Crucibles.—Silver crucibles are but rarely used, save for the fusion of potassa, soda, and for the preparation of caustic baryta from the nitrate. For most operations they are well replaced by platinum. For acid substances their use is improper. The spirit or gas lamp is the heating apparatus, but the heat must not be too high nor of too long duration, for the silver is apt to become brittle in spots as it assumes a crystaline form under the influence of long continued red heat.

Platinum Crucibles.—Platinum crucibles are of more general application than those of any other material. They are very tough and infusible at any heat that can be obtained from the gas or spirit-lamp, the almost exclusive means employed for that purpose. As they are liable to become rough at high furnace temperatures, they should, when exposed to such influences, be inserted in an earthen crucible, and surrounded by a bed of magnesia.

Their strong resistance to the action of chemical re-agents renders them indispensable in many operations, which it would be difficult otherwise to perform. They vary in size from a fluidrachm to three or more fluidounces capacity, the latter



being as large as is necessary for any purpose in a private laboratory. Their form is shown in Fig. 164.

The crucibles intended to be heated over the lamp must be of very thin metal, so that they can be weighed, as is often necessary, in a delicate balance. To give strength, however, the bottom must be thicker than the sides. Two of the smaller sized will be found more useful than one of the larger. In analyses a half ounce crucible is indispensable for the IGNITION, of filters.

The cover is, as seen in the figure, slightly convex exteriorly, and ledged around the circumference: this form is convenient when the vessel is to be entirely closed when heated; but in certain operations in the wet way it is reversed, so that the convex side may look inwardly and return any particles of its contents that may be projected upwards, by a too sudden or intense elevation of temperature. The pin running through its centre is the knob by which it is handled when cold with the fingers, when hot with the tongs, Figs. 114, 127.

For evaporation the crucible takes the form of a capsule, as is seen in Fig. 165, which represents one with a lip and handle. Fig. 165.

Unless the crucibles are made of perfectly pure metal, and are hammered out instead of turned, their power of enduring strong heats and resisting the action of

strong heats and resisting the action of chemical reagents will be impaired. The blisters and flaws which appear, after use, are owing to impurity and bad workmanship, and are to be removed by the force of a small hammer.

When the crucible becomes cracked or perforated it can be repaired by welding on a layer of platinum sponge, but it is far better to have it melted up and remodeled by a manufacturer. (J. Bishop, Philadelphia.)

Boiling or hot water loosens adherent saline matters, and fused borax or muriatic acid will remove all stains which do not disappear by rubbing with sand or pumice stone. The use of sharp pointed instruments will be apt to injure the crucible.

The experimenter himself can in any emergency readily form a crucible out of platinum foil by shaping it with the thumb or a small hammer, in a hemispherical cavity made in a board for the purpose.

Berzelius (*Traité de Chimie*, vol. viii.) gives the following instructions as to the manner of using platinum crucibles.

"Dry fusion should never be effected in platinum crucibles: 1st. Caustic alkalies, nitrates of lime, baryta or strontia and alkaline nitrates always attack the platinum. Alkaline sul-



phurets or sulphates with charcoal are still more injurious. Metals when heated to their melting points alloy with it, and hence lead, tin, antimony, &c., should never be even moderately heated in it. Even their oxides, especially those of copper, lead, bismuth and nickel, reduce at a high heat by contact with platinum, particularly if charcoal is present, the two former at a lower temperature than the latter. Gold, silver, copper and others can be reddened, but not melted in platinum. Phosphorus or phosphoric acid and carbon readily attack it. Sulphate of lead may be burned off in it with care, but for the chloride, porcelain should be used.

Silica may be ignited in platinum, but it combines with silicium at a heat beyond redness, and therefore they should always be encased when heated in the fire, otherwise if in contact, it will abstract it from the coals.

Nearly all liquids may be heated in platinum, except they contain chlorine, bromine, iodine or nitro-muriatic acid.

For the fixed alkalies, gold is preferable to either silver or platinum, upon which they have a more or less corrosive action.

Directions for Heating Crucibles.—All the larger and coarser crucibles are heated in FURNACES. Their proper position, a vertical one, is in the centre of the grate upon a slight elevation. Ignited coals are placed at the bottom of the grate and covered with alternate layers of unlit coke and charcoal, of nut size, until the crucibie is surrounded up to the level of its top with fuel. When the crucible is to be strongly heated, it should be covered and the fuel heaped over its top. In all cases the fire must be gradually raised and steadily kept up, and the furnace only opened when fresh additions of coal are necessary, as it is important that there shall be no variation of the temperature in its interior.

After the completion of the operation, the crucible should be allowed to cool with the furnace, or if taken out immediately, placed upon a brick or bed of warm sand, otherwise a too sudden change of temperature will cause its fracture. The furnace tongs, Fig. 112, are conveniently shaped for this purpose.

As it is occasionally necessary to poke the fire in order that the fuel may settle previous to fresh additions, it will be well to give the crucible a firm position upon a stand for the purpose—the half of a fire brick for instance, so that in the
## FUSION OF SUBSTANCES UNALTERABLE BY HEAT OR AIR. 199

settling of the coal, there may be no risk of its being upset. When by intense heat its bottom has become welded to the brick, the latter can very readily be detached by a gentle tap of the poker.

Most of the common crucibles serve only for a single operation.

Covers may be made by inverting a smaller crucible over the top; or better, by making a dough of Stourbridge clay, and luting it on. The crucible in the latter case must not be heated until the cover has dried. These lids have a tendency to retard volatilization and are necessary to prevent the entrance of falling particles of coal and ashes. For the escape of gaseous matter a small perforation in the centre of the cover is necessary, but in intensely hot fusions all other openings must be closed with LUTE.

The smaller metallic crucibles are almost exclusively heated over LAMPS. They are supported upon wrought iron rings, Figs. 127, 133, the diameter of which may be reduced when necessary, by the use of the wire triangles, fig. 134, of the required size.

If the crucibles are very small they may be heated by the mouth blow-pipe. For the larger an argand spirit or gas lamp, Fig. 27, is needed. To hasten the process or to increase the temperature, the table blow-pipe, Figs. 30, 127, is convenient, as it gives a powerful blast.

The use of the jacket, Fig. 120, is an additional means of still further economizing and increasing the power of the flame. It also diminishes the loss of heat from the crucible by radiation, especially when the latter is covered. In charging the crucibles, the contents should be concentrated into as small a space as possible, and any adherent particles should be brushed from the sides with a feather. When the crucibles are emptied of their fused contents, the melted matter may be made to flow upon a smooth and clean slab of marble, iron or other proper material—great care being taken that it does not come in contact with any moisture or damp substance.

Fusion of Substances unalterable by Heat or Air.—This class comprises a very large number of substances, among which are the noble metals, &c. The crucible employed should be kept covered as well whilst cooling as heating, and the refrigeration must be gradual or the molten matter may

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spirt. There are other metals again, for instance, zinc, lead, tin, antimony, and bismuth, which at high temperatures oxidize readily upon exposure. In such cases it is well, in addition to keeping the vessel closed, to cover the fluid mass with a layer of powdered charcoal.

When a metal is in process of fusion it is imprudent to make fresh additions without having first heated the material to be added, for the sudden entrance of cold or damp matter into the hot fluid mass will cause the ejection of particles, and perhaps serious inconvenience.

In the manufacture of alloys the metals should be well incorporated by occasional stirring. When iron, and indeed manganese, cobalt, nickel and chrome, are being exposed to high degrees of heat, the crucibles must be free from carbonaceous matter, otherwise a combination may ensue at high temperatures.

Fusion of Substances alterable by Heat.—For the treatment of substances which melt below  $212^{\circ}$  F., the water-bath is convenient. The fusion of substances such as wax, resin and fat, immiscible with that liquid, may be facilitated by the direct application of boiling water, as they can be readily removed from the surface to which they rise, with a ladle or syphon whilst hot, or in a mass if allowed to cool.

Substances requiring a temperature at or below 550° for their fusion, may be melted in an oil-bath.

Alloys containing volatile metals should be heated as quickly as possible.

Certain substances which volatilize at low temperatures require to be fused in closed vessels. Iodine and arsenic are examples.

A tube of glass, porcelain or metal, according to the nature of the substance, is the best form of apparatus for this purpose. It should be rounded at one end, and after the introduction of the substance, closed at the other over the blowpipe.

The tube must be heated throughout its length.

Fusion of Bodies alterable by Air.—This class of substances is melted in seclusion, the air being shut out by means of an intermedium of liquid, powder, or fusible matter. Thus potassium is liquefied under naphtha; phosphorus under water, and certain other substances in powdered charcoal.

# FUSION OF DIFFICULTLY FUSIBLE SUBSTANCES.—IGNITION. 201

The covers of the crucibles in these cases must be tightly luted so that all openings may be closed.

Fusion of difficultly fusible Substances.—All substances which resist the fusing power of furnaces, are to be subjected to the more intense action of the HYDRO-OXYGEN BLOW-PIPE.

# CHAPTER XV.

## IGNITION.

SUBSTANCES frequently require to be ignited to redness either as the sole process of their preparation, or as a preliminary step to subsequent operations.

Ignition of Filters.—In analyses, the filters containing the insoluble or precipitated substances which are to be estimated are ignited or "burned off" to expel carbonaceous and volatile matters, before being weighed. The implements for this purpose are porcelain or platinum crucibles, either having their appropriate application.

As it is necessary that the filter should be wholly or par-tially dry, it must be carefully removed from the funnel, so as not to lose a particle of its contents, compressed between the folds of bibulous paper, and, further, dried in a capsule over a sand or water bath, or in a drying stove (DE-SICCATION), at a temperature of about 200° F. or less. The dried filter is then to be transferred to the crucible which has been previously weighed. The transfer must be made without the loss of the least particle, and for this purpose the crucible may be placed upon a sheet of glazed white paper, so that any particles that accidentally fall may be preserved. The filter should be placed in the crucible with its apex upwards, after having been freed as much as possible from the adherent precipitate by gently rubbing the sides together between the thumb and forefinger. The force used for this purpose must not be sufficient to abrade the paper, otherwise the matter will reach the fingers, and a loss thus be occasioned by adherence. The crucible is then heated cautiously and gradually over the spirit or gas lamp, Fig.

127, the flame of which may be urged by the blast. For the first few moments the vessel should remain covered, for fear of loss by decrepitation, but as soon as it becomes red-hot the lid may be wholly or partially removed, and the crucible slightly inclined, as at Fig. 27. This position allows the free admission of air and the complete and rapid incineration of the filter. This done, the cover is replaced, the crucible allowed to cool, and then weighed. The weight of the crucible and that of the ashes of the filter, which latter has been previously determined by the incineration of filters of different sizes, deducted from the total weight, gives the weight of the ignited precipitate.

When substances are to be ignited for the determination of their hygroscopic, volatile, or organic matter, the heat of the lamp should be gradually applied without the blast, and, for the former purpose, only to the production of a dull red heat. In these instances, the crucible should be weighed first, so that the loss sustained by a given weight of its contents during ignition, may be ascertained in one weighing merely by subtracting the weight of the crucible and contents after ignition from the combined weight of the two before the same process. The loss gives the amount of volatile matter.

In analyses of coals, the moisture can be determined by heating the crucible in a hot sand-bath, or very gently over a low flame. After the loss thus occasioned is determined by weighing, the amount of carbon may be ascertained by subjecting the crucible and contents to a much higher heat.

When substances are to be exposed to heat, the crucible and contents must likewise be weighed separately before ignition. The loss of weight gives the amount of volatile matter driven off. The ignited matter can then be removed from the crucible by hot water alone or acidulated.

Scoriæ may be removed from platinum crucibles by covering them with a paste of borax and carbonate of soda, heating them to redness, and when cold, dissolving out the saline matter with boiling water. A repetition of the process is necessary to brighten the crucible perfectly if it had been very dirty.

*Ignition of Bodies in Vapors.*—If it be desired to heat a fixed substance in the vapor of any body, which is solid or liquid at ordinary temperatures, the latter may be put into a tube closed at one end, or into a small flask with a long neck, and then be heated until it is wholly vaporized. The substance is to be introduced into the tube, and heated in the vapor at any desired temperature. Thus, to show the affinity of sulphur for copper, the former is heated until its vapor fills the whole flask, when slips of copper foil let down into it immediately ignite on combining with the sulphur. When a tube is used it may be held in any inclined position, but a flask should be nearly vertical.

Ignition with Fluxes.—Fluxes are certain substances usually saline, mixed with other bodies in order to promote their fusion or decomposition by heat, and, to render them more soluble in water and acids. All ignitions with fluxes in experimental operations are performed in crucibles over the spiritlamp or furnace fire, and for the fluxions of those substances in which there is no reducible metallic oxide, platinum is by far the best material.

The process is particularly useful in the analysis of the sulphurets of alkaline earths, of many silicates and other obstinate compounds and also in metallic operations.

The principal objects of fluxing are:-

1. "To cause the fusion of a body, either difficultly fusible, or infusible by itself.

2. To fuse foreign substances mixed with a metal, in order to separate the latter by its difference of specific gravity.

3. To destroy a compound into which an oxide enters, and which prevents the oxide being reduced by charcoal. The silicate of zinc, for instance, yields no metallic zinc with charcoal, unless it be mixed with a flux capable of combining with the silica.

4. To prevent the formation of certain alloys, and consequently the combination of some metals with others, as in the case of a mixture of the oxides of manganese and iron with a suitable flux, the iron is obtained in a state of purity, whereas if no flux had been added, an alloy would have been obtained. Gold and silver can be separated from many other metals by means of a flux.

5. To scorify some of the metals contained in the substance to be assayed, and obtain the others alloyed with a metal contained in the flux, as gold or silver with lead.

6. And lastly, a flux may be employed to obtain a single button of metal, which otherwise would be obtained in globules." Fluxes should always be pulverized, and whether mixed directly with the substance before ignition, or added gradually to the crucible during the process, ought rather to be in excess than in deficiency. In the fluxion of silicates the material and flux are incorporated together before being transferred to the crucible.

When the mixture is of a frothing nature, it is best to add it to the crucible piecemeal and to heat it gradually, so as to save the loss caused by ejection of particles. After the whole has been placed in the crucible, the heat may be raised and maintained until perfect fusion and the completion of the process.

Fluxes are divided into non-metallic and metallic fluxes.

NON-METALLIC FLUXES. — (Berthier, *Essais par la voie* Sche.)—*Silica* is employed frequently to cause the fusion of some gangues in assays made at an elevated temperature. Silica combines with all the bases, and forms with them bodies termed silicates, which are more or less fusible.

Lime, Magnesia, and Alumina.—It is known that no simple silicate is readily fusible, so that lime, magnesia, or alumina are employed, according to circumstances, to reduce a simple silicate to such a condition that it will readily fuse in an assay furnace. Sometimes, to attain this end it is requisite to use all the above-mentioned earths, for experience has proved that as a general thing a mixed or double silicate fuses more readily, and flows freer than a simple silicate.

Baryta.—Hydrate of baryta fuses at a low red heat, and without loss of its water of crystallization, and for the first reason is preferable to either the carbonate or nitrate. It is used in silver or platinum crucibles, and when silicates are to be tested for alkalies. The silicates of baryta, however, fuse with difficulty, and are sluggish.

Glass is a very useful flux in certain iron assays. The kind employed must contain no lead.

Boracic Acid.—The native boracic acid, after fusion and pulverization, is to be employed whenever the use of this acid is indicated. It ought to be kept in well-stopped bottles.

Boracic acid has the property of forming with silica and all the bases very fusible compounds, and is from this cause a very universal flux. Nevertheless, there is an inconvenience attached to its use; it is very volatile, so that sometimes the greater part employed in an assay sublimes before it has had time to perform its office. Borax, Biborate of Soda, is an excellent and nearly universal flux, because it has the property of forming, like boracic acid, fusible compounds with silica and nearly all the bases, and is preferable to that acid because it is much less volatile.

It may be used at a high or low temperature. In the first case, it is employed in the assay of gold and silver because it fuses and combines with most metallic oxides, or in obtaining a *regulus*, that is to say, to separate the metals, their arseniurets and sulphurets, from any stony matter with which they may be mixed, because this salt is neither oxidating nor desulphurating. In the second case, it is employed in the assay of iron and tin ores, as in the presence of charcoal it retains but traces of their oxides, and, indeed, much less than generally remains with the silicates.

When borax is heated it fuses in its water of crystallization, and undergoes an enormous increase of volume; at a higher temperature, it fuses and forms a transparent glass, which becomes dull on the surface by exposure to air. Only the fused vitrified borax ought to be used in assays. It must be reduced to powder, and kept in well-closed vessels.

Fluor Spar, Fluoride of Calcium, is rarely employed in assays, but in certain cases is an excellent flux, especially where sulphates are present, with many of which it forms very fusible compounds. The best proportions are about equal equivalents of the spar and the anhydrous sulphates of alkali, lime, and oxide of lead: but for the sulphate of baryta, two eqs. of the spar for one eq. of the sulphate.

It likewise assists in fluxing silicates, partly by direct union with them, and partly by yielding fluosilicic gas, and leaving lime to unite with silica.

Carbonate of Potash and Carbonate of Soda.—It has been already proved that they possess oxidating and desulphurating power; they will now be considered as fluxes.

They are decomposed in the dry way by silica and the silicates, with the separation of carbonic acid. The presence of charcoal much facilitates this decomposition.

The silicates of potassa and soda fuse readily and flow freely.

They form fusible compounds with the greater part of the metallic oxides; in these combinations the oxide replaces a certain quantity of carbonic acid; but these compounds are not stable, they are decomposed by carbon, which reduces the oxide, or by water, which dissolves the alkali. On account of their great fusibility, the alkaline carbonates can retain in suspension, without losing their fluidity, a large proportion of pulverized infusible substances, as an earth, charcoal, &c.

The alkaline carbonates ought to be deprived of their water of crystallization for assaying purposes; in fact, it would be better to fuse them before use. They must, in all cases, be kept in well-stopped vessels.

They may be used indifferently, but carbonate of soda is to be preferred as it does not deliquesce.

À mixture of both is far preferable to either alone, and moreover requires a lower heat for its fusion. The proper proportions are ten parts of effloresced carbonate of soda and thirteen parts of dry carbonate of potassa. The two are to be intimately incorporated by trituration and the mixture kept in stoppered bottles. This flux is the one of most general application.

The alkaline carbonates of commerce always contain sulphates and chlorides. In ordinary cases, this causes no inconvenience, but there are circumstances under which the presence of sulphuric acid would be injurious.

Carbonate of potash can readily be procured free from sulphate and chloride by means of nitre and charcoal, as follows:—Pulverize roughly 6 parts of pure nitre, and mix them with 1 part of charcoal; then project the mixture spoonful by spoonful into a red-hot iron crucible. The projection of each spoonful is accompanied by a vivid deflagration, and carbonate of potash is found in a fused state at the bottom of the crucible; it must be pulverized, separated from excess of charcoal, and kept in a dry state for use.

Carbonate of soda may be obtained in much the same way, substituting nitrate of soda for nitrate of potash; or by repeatedly crystallizing the carbonate of commerce.

Nitrate of Potash.—The presence of silica or silicates much assists its decomposition. It is used as an oxidizing agent, the potash resulting from its decomposition acting as flux. To prevent violent action and ejection of particles of matter, its addition to the crucible must be careful and gradual. Nitre is also employed in some instances as a substitute for nitrate of ammonia for effecting the rapid and perfect combustion of organic substances.

Common Salt, Chloride of Sodium, was much recommended

by the older assayers, either mixed with flux, or a certain quantity placed above it, for the purpose of preserving the substances beneath from the action of the atmosphere, or to ameliorate the action of such bodies as cause much ebullition. It is very useful in lead assays. When it is used, it must be previously pounded and heated to dull redness in a crucible to prevent its decrepitation.

Black Flux and its Equivalents.—Black flux is both a reducing and fusing agent. It is a mixture of carbonate of potash and charcoal in a minute state of division. It is much employed, and very serviceable. It is prepared by mixing 2 parts of argol with 1 part of nitre, placing the mixture in an iron vessel and setting it on fire by a burning coal or red-hot rod. When the combustion is finished, the substance is pulverized and sifted whilst yet hot, and kept in well-stopped jars, as it rapidly absorbs moisture from the atmosphere.

Black flux is much used in lead and copper assays; but as it boils up greatly at the commencement of the operation, the crucible must not be more than two-thirds full.

It can be readily imagined that, as it fuses and reduces at the same time, the relative proportions of alkaline, carbonate, and charcoal ought to vary according to the nature of the substance acted upon; and it is often expedient to employ the greatest possible proportion of alkali to obtain the largest yield of metal. Black flux may be obtained richer in carbon by mixing 1 part of nitre with  $2\frac{1}{2}$  or three parts of argol.

Common black flux contains 5 per cent. of charcoal. The flux prepared with  $2\frac{1}{2}$  of tartar or argol to 1 of nitre, contains 8 per cent., and that with 3 contains 12 per cent. of charcoal.

Black flux can be replaced by anhydrous or dry carbonate of soda mixed with some reducing agent. When charcoal is employed it must be reduced to a very fine powder; in fact, it ought to be levigated.

The three following fluxes are very useful:

 Carbonate of Soda
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 .
 .
 94
 88
 816

 Charcoal
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 .
 .
 6
 12
 184

The second is very nearly equivalent to sodium and carbonic acid, and the third to sodium and carbonic oxide; but it must be observed, that whatever precautions be taken, these mixtures never become so liquid as black flux, because the charcoal tends very much to separate and rise to the surface. Instead of charcoal, it is preferable to use sugar or starch to make a flux equivalent to black flux with carbonate of soda; the mixture must be made most intimately.

Cream of tartar, carbonized by a semi-combustion until it is reduced to half its weight, is a very good substitute for black flux: it contains about 10 per cent. of charcoal.

Argol, Cream of Tartar, Bitartrate of Potassa.—When bitartrate of potassa is heated in a covered crucible, a rapid decomposition takes place, accompanied by a disengagement of inflammable gases; the substance agglomerates, but without fusing or boiling up. The residue is black, blebby, and friable, and contains 15 per cent. of carbon when produced from rough tartar or argol, and 7 per cent. from cream of tartar.

These reagents produce the same effects as black flux, and possess more reducing power, because they contain more combustible matter; but this is an inconvenience, because the excess prevents their entering into full fusion when the substance to be assayed requires but a small proportion of a reducing agent. They can be used with success in assays requiring much carbonaceous matter.

Bisulphate of Potassa is a convenient flux for several minerals, such as for those highly aluminous (Rose), for chromic and other similar ores (Booth).

Salt of Sorrel, Binoxalate of Potassa, when heated is decomposed. It decrepitates feebly, and during its decomposition is covered with a blue flame; it at first softens, and when fully fused, is wholly converted into carbonate. When the oxalate is very pure, the resulting carbonate is perfectly white and free from charcoal; but very often it is spotted with blackish marks. It has no very great reducing power.

Cyanide of Potassium acts powerfully both as a reducing and desulphurizing reagent, and is a very useful flux in small assays. According to Liebig it has the advantage over the potassa salts with vegetable acids, of not carbonizing the metal, for the salt changes at the expense of the metallic oxide into cyanate of potassa. If the metallic oxide predominates, the rest will be reduced by the cyanic acid without separation of carbon.

White, or Mottled Soap is a compound of soda with a fat acid. When heated in close vessels it fuses, boiling up considerably, and during its decomposition gives off smoke and combustible gases, and leaves a residue composed of carbonate of soda with about 5 per cent. of charcoal. Of all reducing agents soap absorbs the greatest quantity of oxygen, and as the residue of its decomposition by heat affords but little charcoal, it has the property of forming very fluid slags. Nevertheless, it is rarely employed because certain inconveniences outweigh its advantages. These inconveniences are, its bubbling up and its extreme lightness. It also requires to be rasped, in order to mix it perfectly with the substances it is to decompose, and it then occupies a very large volume, and requires correspondingly large crucibles. There are nevertheless cases where it may be used with advantage by mixing it with other fluxes.

All those fluxes containing alkaline and carbonaceous substances are reducing and desulphurizing, besides acting as fluxes, properly so called; they also produce another effect which it is useful to know, viz : they have the property of introducing a certain quantity of potassium or sodium into the reduced metal. This was first pointed out by M. Vauquelin.\* He found that when oxide of antimony, bismuth, or lead was fused with an excess of tartar, the metals obtained possessed some peculiar characters, which they owed to the presence of several per cent. of potassium.

METALLIC FLUXES—*Litharge and Ceruse.*—These bodies always act as fluxes, but at the same time often produce an alloy with the metal contained in the ore to be assayed. Ceruse produces the same fluxing effect as litharge. The litharge is the better flux, and is very useful in a great number of assays.

It fuses readily with the oxides of iron, copper, bismuth, antimony and arsenic, sulphate of lead and the silicates, in the proportion of 2 to 5 parts of litharge to 1 part of the substance to be fluxed; other oxides require a larger amount of litharge. Its action is that of promoting fusion, reducing an oxide and desulphurizing a sulphuret.

Glass of Lead, Silicate of Lead.—The silicates of lead are preferable to litharge in the treatment of substances containing no silica, or which contain earths or oxides not capable of forming a compound with oxide of lead, excepting by the aid of silica. It may be made by fusing 1 part of sand with 4

\* Annales des Mines.

parts of litharge; if required more fusible, a larger proportion of litharge must be added.

Borates of Lead.—The borates of lead are better fluxes than the silicates when the substance to be assayed contains free earths; but in order to prevent them swelling up much when fused, they must contain an excess of oxide of lead. The borate of lead containing .9056 of oxide of lead and .0944 of boracic acid, is very good. Instead of borate of lead, a mixture of fused borax and litharge may be employed; it is equally serviceable.

Sulphate of Lead is decomposed by all silicious matters and by lime, so that when these substances are present litharge is produced, which fluxes them.

Oxide of Copper is rarely used as a flux for oxidated matters, but is sometimes employed in the assays of gold and zinc to form an alloy with those metals. In this case a reducing flux must be mixed with the oxide. Metallic copper may be used, but is not so useful, as it cannot be so intimately mixed with the assay.

Oxides of Iron are good fluxes for the silicates. They are, however, rarely employed for that purpose; they are more often used to introduce metallic iron into an alloy to collect an infusible, or nearly infusible metal, by alloying it with iron, such as manganese, tungsten, or molybdenum.

CALCINATION.—The separation (in a dry way) of volatile from fixed matter, by heat, is termed calcination. The process is applicable

To the expulsion of water from salts, minerals, coals and other substances.

" carbonic acid from certain carbonates. 1

" arsenic and sulphur from cobalt, nickel and other sulphuretted compounds.

" bituminous matter from coals, and certain minerals and ores.

To the ignition of quartz and silicious minerals to promote their disintegration (p. 77).

For the purpose of expelling the combined water of argillaceous minerals, and of thus rendering them more obstinate to the solvent action of acids and reagents.

If the substance under process is organic, its calcination in a close vessel by a medium heat usually effects only partial decomposition, the gaseous matter generated escaping through interstices and the fixed components remaining with a portion

66

66

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66

66

of unaltered carbon. Performed in this manner, the process takes the name of *coking*, familiar instances of which are the formation of coke by distilling coal in closed retorts, the manufacture of charcoal from wood, and of bone black from bones.

By increasing the temperature and admitting the air, the whole of the alterable and volatile matter is expelled, the fixed matter remaining as ashes. The process is then styled *incineration*, and in this way the coke, charcoal and ivory black, obtained as above directed, may be entirely reduced to their incombustible portions or ashes.

Calcination is effected in platinum spoons or crucibles, in delicate experiments, over a spirit lamp; but in large operations a furnace is required, and the containing vessels are crucibles of either metal or earthenware, according to the nature of the substance to be heated, though the latter are often unsuitable for temperatures above a red heat.

When the operation is finished, the crucible should be taken from the fire and allowed to cool gradually. The cover is then to be lifted off and the contents taken out with a spatula, and the portions adhering to the sides removed with a feather.

If the substance undergoing calcination is fusible, it is necessary when quantities are to be ascertained, to weigh both the crucible and contents before ignition, so that the amount of volatile matter driven off may be expressed by the weight lost in heating. Water alone or acidulated, with the aid of heat generally removes the calcined matter from the crucible.

A body decrepitating by heat should be powdered before being subjected to the process of calcination, and the temperature should be raised slowly and gradually, otherwise when the crucible is not covered, a loss may result from the ejection of particles.

To avoid contact with the generated vapors or with the atmosphere, which to some substances act as reducing agents, the crucible should in such cases be covered, and if tightly luted perforated with one or more small holes for the escape of vapor.

Roasting (as the term is generally used) is a kind of calcination to which many ores are submitted before their final reduction to the metallic state, for the purpose of expelling ingredients which would either delay that process or be injurious to the metal when extracted. In this way water, carbonic acid, sulphur, selenium, arsenic, and sometimes other substances, are driven off from the ores containing them. The term is also applied to other processes, among the most important of which is that of the exposure to heat and air by which metals become altered in composition. Thus, copper becomes oxidized, and antimony and arsenic acidified by union with oxygen.

Roasting is always effected in broad, shallow open vessels, so that the air may have free access; and in order to promote the absorption of oxygen or the escape of the volatile substance, the surface of the body to be heated should be increased by previous pulverization, and it should be constantly stirred during the operation so as to present as many points of contact as possible. The most suitable vessel is a baked earthenware saucer or capsule placed in a muffle or upon the bars of a calcining furnace. Sometimes a crucible is used, and then the position of the vessel in the furnace should be slightly inclined on one side. In either case the vessels should be heated to dull redness previous to receiving their charge.

That species of roasting termed *deflagration* is effected by rapidly heating the substance to be oxidized, together with some additional body as an oxidizing agent, as a nitrate or chlorate for instance. The powdered mixture is added portionwise to the crucible previously heated, and maintained at redness during the operation. The vivid and sudden combustion which ensues modifies the composition of the original substance and increases its amount of oxygen at the expense of the addendum. Thus for instance, sulphuret of arsenic is deflagrated with nitre to produce arseniate of potassa, titanium and certain other metals to be transformed into oxides.

Deflagration is also used as a means of detecting the presence of nitric or chloric acids. For this purpose the suspected substance is to be heated with cyanide of potassium, in a small platinum spoon. If deflagration ensues it is a test of the presence of one of them, or a compound of one of them.

The crucibles may be of clay or metal according to the nature of the substance to be heated. The roasting of substances for the expulsion of organic matter may be effected in platinum vessels, provided the heat is not carried sufficiently high to produce fusion of the substance being roasted. The heat must, at first, be very gradually applied, and at no time be made great enough to fuse or agglutinate the material, otherwise the process will have to be suspended in order to repulverize the matter. Proper care at the commencement will obviate the necessity of this additional trouble. When the heat has been cautiously raised to redness and all liability of fusion is over, the fire may be urged to the production of a yellowish red or even white heat, so that the expulsion of volatile matter may be complete.

Roasting operations which disengage deleterious or disagreeable fumes should be carried on in the open air or under a hood, and when the volatile matters are valuable they may be condensed as directed in DISTILLATION and SUBLIMATION.

Decrepitation, which frequently occurs and occasions loss by ejections of particles of the mixture, is owing to the sudden vaporization of the water of crystallization, which in finding vent scatters the confining substance with a crackling noise. To prevent this loss, the crucible should be loosely covered until decrepitation ceases.

REDUCTION.—This operation is employed for the separation of metallic bases from any bodies with which they are combined; but is generally confined to the extraction from an oxide—that being the kind of combination most commonly met with. The combined action of heat and certain reagents is required to effect this result, the temperature varying with the nature of the substance to be reduced.

The most usual reducing agents are charcoal and hydrogen gas. Tallow, oil and resin are sometimes used, but being easily decomposed they are dissipated before entire reduction has occurred. Sugar and starch are also occasionally employed. We shall, however, confine our remarks to the two principal articles.

. Reduction by Charcoal.—Charcoal is used for this purpose in two ways, either in powder and directly mixed with the substance, or as a lining coat to the crucible in which the reduction is accomplished. The first mode is objectionable, because the excess of coal which is required to be used interferes with the agglomeration of the particles of reduced metal. Whenever it is adopted, the quantity of coal dust to be added, which must be sufficient to transform all the oxygen of the oxide into carbonic acid, can be determined by calculation. This amount is then mixed thoroughly with the oxide previously powdered, and is transferred to a crucible, taking care to place the charge in the centre and to cover the contents with a layer of the dust. The whole is then to be subjected to the heat of a furnace, assisted if necessary, by a blast. The reduction in this way, the most convenient for large quantities, is rapid and complete, but the metallic residue is often mixed with coal dust.

In general the mere contact of carbon is sufficient to effect reduction, and consequently the inconvenience of the above plan may be avoided by the use of a brasque or crucible lined interiorly with charcoal. An earthen crucible is very readily brasqued as follows :- A mixture of three parts of charcoal dust, and two parts of powdered clay, is mixed with water and kneaded into a plastic dough. The bottom of the crucible is then covered with this dough, and a wooden cylindrical core of diameter equal to that required for the cavity, is inserted in the centre and surrounded with more of the same dough, which is compressed with the fingers at each addition so as to make the whole as compact as possible. The core is then to be carefully withdrawn, and the crucible placed aside to dry. A platinum crucible, which is as applicable as clay for certain operations, can be brasqued in the same way. Some operators use the coal dust without clay, and moisten it with water or The crucibles should be free from external fissures to oil. prevent access of air, and must always be covered when The reduction by this plan is slower than by the heated. first mode, and requires a higher temperature, but the metal as procured is cleaner.

The powdered oxide is placed in the cavity in sufficient quantity to fill it, then compressed with the fingers and covered with a layer of coal dust. The cover being luted upon the crucible the whole is to be heated in a blast furnace. The reduction proceeds from the surface, that part of the oxide next to the charcoal being first acted upon. The time required depends upon the nature of the oxide, the degree of temperature and the quantity under process : sometimes, particularly when the metals are very fusible, the reduced particles collect in a clean lump at the bottom of the crucible, and are easily removable when cold, with the finger or spatula. Others again, more refractory, form a very friable lump of metallic powder.

Reduction by Hydrogen .- This mode, which is much used in

#### **REDUCTION APPARATUS.**

analyses, consists in passing a current of hydrogen gas over the metallic oxides heated to redness in a glass, or better, porcelain tube, and is equally applicable to some chlorides and other compounds. The arrangement of the requisite apparatus is shown in Fig. 166. A is a flask for the disengage-



ment of hydrogen gas, by the action of dilute sulphuric acid upon zinc, the funneled tube a being for the ingress of the acid. The disengagement tube b is bent at right angles and bulbed midway in its horizontal arm. The bulb is to be furnished with a plug of raw cotton for the condensation and retention of any aqueous vapor that may pass over. This tube is joined hermetically to another short tube c by means of an India rubber connection.\* The connecting tube is made

\* The use of India rubber as a material for forming flexible joints is one of the most important aids in chemical manipulation, as is shown by a reference to many pieces of apparatus. Its property of readily uniting at freshly cut surfaces, its flexibility, its ready and close adhesion to surfaces, and power of resisting the action of corrosive vapors except those of chlorine, sulphuric and nitrie acids and a few others, render it peculiarly excellent for many mechanical purposes of the laboratory. Tubes of any shape and size, according to the form and dimensions of the parts of apparatus to be connected, are to be fashioned out of it with almost equal facility. For the transmission of corrosive vapors or gases they should have an outer layer, the seam in which must be directly opposite to that in the tube which it invests, so as to ensure perfect tightness. Prof. Booth uses the India rubber pipe, made by Goodyear as conduits for steam in boiling corrosive liquids of sheet caoutchouc about one-twelfth of an inch in thickness.



A piece of the required length of the tube and twice the intended width is cut out and wrapped around a cylindrical glass rod, d, Fig. 167, of diameter very nearly as great as that for the tube to be formed. The ends are then brought closely together by compression between the thumb and fingers as at a, and the excess

removed, close to the surface of the rod, with a pair of clean sharp scissors. The freshly cut edges being further pinched together throughout the length of the tube, form a close, air-tight, scarcely perceptible joint. The rod is then to be withdrawn and the tube thus formed carefully drawn over the end of one of the glass tubes to be connected, so as to form an extension for the reception of the end of the other. The two ends should approach each other almost to contact, a minute interval being necessary to afford the necessary flexibility. This junction pipe is fastened to the surface of the tube by fine twine wrapped or tied around each of its ends, as shown at x, Fig. 166.

The gas bottle thus fitted is connected, by means of a perforated cork with the drying tube d, filled with lumps of dried chloride of calcium. At the opposite end of the drying tube

by that agent, and gives it consistence with flexible lead pipe, which he covers externally and internally. A better frame work would be a spiral coil of wire. The tubing made of canvas imbued with caoutchouc is less durable, and does not admit of such general application.

Before forming the tube above mentioned, it is better to warm the caoutchouc, by which its flexibility is increased and its cut surface made to adhere more readily and closely. The scissors cut more freely when previously moistened. These flexible joints not only relieve the apparatus of stiffness and consequent liability to fracture, but enable the operator to adjust it more rapidly and satisfactorily than he could possibly do without them. A little practice upon shreds will give great proficiency in the art of forming India rubber tubes and joints.

India rubber for this purpose is now made by Goodyear, New York, who sells it in sheets of various sizes. Gas bags are also made of caoutchouc. The larger sized, pp. 171, 173, are to be procured from the manufacturer. Smaller ones, for nice purposes, may be readily made from the rubber bottles of the shops. One of uniform thickness, and as free as possible from indentations and imperfections, is softened in boiling water or by exposure for several hours to the vapor of ether, and then adjusted upon a stop-cock with a syringe attached. The air is then to be injected slowly so that the expansion of the bag may be gradual and uniform throughout all its parts. e, is another tube with a bulb blown in its centre for the reception of the substance to be reduced, and in which it is heated by the flame of a spirit lamp. This tube, like the other, is annexed by elastic joints to the short tube connected with the desiccating tube through a perforated cork.

This plan, first proposed by Berzelius, was used by him in the synthesis of water, binoxide of copper being the substance employed to abstract the hydrogen, its oxygen forming water therewith.

Hydrogen is a powerful reducing agent, and leaves the metal absolutely pure. At a red or white heat, its action will reduce the oxides of lead, bismuth, copper, antimony, zinc, iron, cobalt, nickel, tungsten, molybdenum, and uranium.

The heat should not be applied to the bulb until it is entirely freed from air, which may be done by allowing the hydrogen to pass over some minutes previously. A disregard of this precaution may cause an explosion from the combustion of a mixture of hydrogen and atmospheric air.

The above apparatus answers very well for decomposing metallic sulphurets by chlorine. It is also applicable for heating solids in gases, and serves for the preparation of chloride of sulphur, of phosphorus, and of many other volatile chlorides. For this purpose it is only necessary to replace the flask A by other suitable generating vessels, and the extreme end of the exit tube by a tubulated retort with its beak bent downwards and leading into the recipient, kept cool by a frigorific mixture.

The tubes for these purposes must be of hard glass and entirely free from lead, and not exceeding a third of an inch in width. The bulbs should be of  $1\frac{1}{2}$  inch diameter. The chlorcalcium tube may be three-fourths of an inch wide.

There are other modes of reduction of less general application, however, than the preceding. Metals may be precipitated in a free state, in some instances, from solutions, by presenting bodies for which their oxygen has a stronger affinity, thus, for example, protosulphate of iron precipitates metallic gold; phosphorous acid mercury; and formic acid or formate of soda, both of these metals, and also silver and platinum, if the liquids containing them in solution are boiled. So also one metal may reduce another if the affinity of the first for oxygen is greater than that of the last. Thus metallic copper throws down mercury, silver, and arsenic from their solutions, and iron precipitates copper.

Metals are also reduced by galvanic action, practical illustrations of which are seen in the galvanoplastic art. All oxides which resist the combined action of heat and charcoal or hydrogen, are reduced by the agency of galvanism.

Reduction by Carbonic Oxide.—Another convenient agent of reduction, employed in the same manner as hydrogen, is carbonic oxide, made on a small scale by the action of oil of vitriol on oxalic acid, and separation of the carbonic acid produced at the same time, by milk of lime. It readily reduces the metallic oxides of nickel, iron, zinc, that of lead at a very low temperature, and that of copper below a red heat. For heating in manufacturing processes, it is made by regulating the admission of air to a deep bed of ignited anthracite or other coals, and driving a blast of air horizontally through the gas as it issues from the fire, all other access of air being prevented. It has in this manner been applied to re-heating and puddling furnaces. Carbonic oxide is doubtless the great reducing agent in large metallurgic operations.

Roasting and Reduction in Tubes.—In very delicate experiments, and particularly when the volatile matter expelled by the heat is to be collected for examination, roasting and reduction are effected in small glass tubes\* closed at one end.

\* Porcelain and Metallic Tubes.—For the reduction of some oxides by contact with gases at furnace temperature, for the decomposition of certain organic matters, such as oils, &c., and for effecting many combinations of gases with solids, the glass tubes are replaced by those of porcelain, iron, or platinum.

Porcelain tubing should be selected with care. It should be straight, perfectly cylindrical, free from defects, glazed internally and as thin as possible. These tubes are adjusted in manner as directed for those of glass, and heated over the furnace, Fig. 103, but as they are not refractory, care must be taken in heating them. It is advisable to give them an exterior coating of fire lute and then dry them. The fire should be ignited and all moisture expelled from the charcoal before they are placed in the furnace, otherwise their fracture may result. It is indispensable, too, that the heat shall be carefully managed, and after the completion of the process the tube must not be removed from the furnace until it has entirely but gradually cooled.

Iron tubes are used for the decomposition of water, potassa and for other operations to which those of glass and porcelain are not adapted by reason of inability to withstand high heat. Gas tubing is the most economical, and can

## ROASTING AND REDUCTION IN TUBES.



The glass must be white, difficultly fusible, and free from lead. The substance is placed in the lower or closed end of the tube,

be had of all lengths and diameters. These also should be covered exteriorly with luting so as to prevent the oxidation of the iron by the fire.

Metallic tubes of small size may be heated over the furnace, Fig. 103, but those of larger dimensions require the use of the furnace, Fig. 88. The circular openings x x, in each side, are especially for the passage of a tube. The grate should be elevated so that the fire may entirely surround it.

Metallic tubes are adjusted to generating and other apparatus by means of metallic couplings, gallows screws, or, in some cases, by fire lute. This latter does not make a secure or tight joint, and is only used in the absence of more convenient means. The ends of the tube should project far enough beyond the sides of the furnace to allow their refrigeration when necessary. The gas may be introduced directly from the generating vessel, or from a caoutchouc bag, or gasometer, merely by adjusting the end of the tube with the mouth or outlet by a suitable coupling. The resultant product may, in like manner, be collected by similar adaptations to the other end.

Fragments of flint or coils of iron or of platinum wire, placed within the tube, increase the points of contact of the contained matter and greatly promote its heating.

As short tubes are occasionally used for effecting the combination of substances alterable by exposure in a hot state, they should, for such purposes, be fitted at the ends with screw plugs to prevent access of air.

Platinum tubes are only used on rare occasions for particular purposes to which those of glass, porcelain, or iron are inapplicable.

which is then inclined and heated over the spirit lamp, as shown in Fig. 174. In this way sulphur and arsenic may be sublimed from certain of their compounds, and mercury from less volatile metals. By leaving the tube open at both ends so as to allow free access of air, many volatile bodies are oxidized and collect, on congelation, in the upper part of the vessel. Those tubes with a bulb blown at their lower end, as shown at 1, 5, in Fig. 168, are most applicable for decrepitating substances.

Below are the several forms of tubes used for the reduction of metals, and particularly the separation of arsenic and mercury from more fixed matter. Any of these forms, or even a small test tube 4 will answer. Berzelius prefers the shape of 1; Rose that of 2; Liebig that of 3; and Clarke that of 5.

The letters  $a \ b \ c$  in 2, and b in 3, indicate the position of the substance to be roasted together with its reducing agent, and d and a in 2 and 3, the rings of condensed volatile matter sublimed by the heat. Berzelius and Rose's, and Liebig's tubes are three inches in length; Clark's two inches. Their diameters vary from  $\frac{1}{16}$ th to a  $\frac{1}{4}$ th of an inch according to the amount of substance to be heated.

# CHAPTER XVI.

#### CUPELLATION.

GOLD and silver are assayed by the agency of heat and litharge in shallow, slightly conical crucibles, Fig. 169, called *cupels*. This process affords these metals free from any debasement with which they may be contaminated; for, when



the alloy is heated together with litharge, all but the precious metals are oxidized; and the oxides thus formed, together with the semi-vitrous litharge, are absorbed by the cupel whilst the nobler metal remains as a button of absolute purity.

Cupels.—They are generally made of bone ash, because that material fulfils better than any other the necessary requirements. It is resistant to the action of the fused oxides of lead and bismuth, and by its porosity facilitates the penetration of the oxides, and at the same time is, when made into shape, strong enough to bear handling without fracture. The cupels used at the mint in this city, are made in a matrix of  $1\frac{3}{8}$ inches diameter. The semi-circular cavity is two-fifths of an inch deep in the centre. This size, however, can be varied and they may be made smaller or larger according to the quantity of matter to be operated upon. Their mode of manufacture is as follows:-Take bones or bone black and calcine them in an open crucible until the expulsion of all animal and carbonaceous matter, which is known by the residue assuming a whitish appearance. Empty the cooled contents of the crucible into clean water, and give it repeated washings in fresh waters to remove all soluble matter; filter and dry. The dried matter is pure phosphate of lime with a minute portion of partially decomposed carbonate.

Take the powder, calcined and purified as directed above, and make it into a paste with water or preferably with beer (Mitchell), in the proportion of 4 lbs. of bone-ash to half a pound of beer. The above mixture is just sufficiently moist to adhere strongly when well pressed, but not so moist as to adhere to the finger or the mould employed to fashion the cupels. The mould, Fig. 170, of polished iron, consists of two pieces, one a ring having a conical opening; the

other, a pestle having a hemispherical end fitting the larger opening of the ring. In order to mould the cupels, proceed as follows: Fill the ring with the composition, then place the pestle upon it and force it down as much as possible; by this means, the moistened bone-ash will become hardened, and take the form of the pestle; the latter must then be forced as much as possible, by repeated blows from a hammer, until quite home. It is then to be turned lightly

round, so as to smooth the inner surface of the cupel, and withdrawn; the cupel is removed from the mould by a gentle pressure on the narrowest end. When in this state, the cupel must be dried gently by a stove; and lastly, ignited in a muffle, to expel all moisture. It is then ready for use.

There are two or three points to attend to in manufactur-

Fig. 170.

ing the best cupels. Firstly, the powdered bone-ash must be of a certain degree of fineness; secondly, the paste must be neither too soft nor too dry; and thirdly, the pressure must be made with a certain degree of force. A coarse powder, only slightly moistened and compressed, furnishes cupels which are very porous, and break on the least pressure, and which allow small globules of metal to enter into their pores—the most serious inconvenience of all.

When, on the contrary, the powder is very fine, the paste very moist and compressed very strongly, the cupels have much solidity, and are not very porous, the fine metal cannot penetrate them, and the operation proceeds very slowly; besides, the assay is likely to become dulled and incapable of proceeding without a much higher degree of temperature being employed.—(Berthier.)

The Process of Cupellation.—In order to protect the cupel from contact with the fire, and at the same time allow a free access of the air, it is when being heated placed in a muffle. The muffle is a refractory vessel of baked fire clay,

Fig. 171.



Fig. 171, arched above, flat bottomed, and pierced near its base with small lateral openings for the passage of the heat. Excepting these apertures, and that at the front for the introduction of the cupels and inspection of the pro-

cess, the muffle is entirely closed. Its dimensions depend upon the size of the cupel and of the furnace in which it is to be heated.

Its position in the furnace (Fig. 98, and D, Fig. 101), must be exactly level, and to protect it from the corrosive effects of volatilized oxides, it may be payed over with a thin paste of bone ashes. The muffle being properly arranged in the furnace, and held firmly in its place by lute, the cupels are





then introduced and the fuel (charcoal) ignited. The lead must be perfectly pure. It can be reduced, for this purpose, from refined litharge. "When the cupels have been exposed for half an hour, and have become white by heat, the lead is put into them by means of the tongs, Fig. 172, and as soon as this becomes thoroughly red and circulating, as it is called, the metal to be assayed, wrapped in a small piece of paper, is added, and the fire kept up strongly until the metal enters the lead and circulates well, when the heat may be slightly diminished, and so regulated that the assay shall appear convex and ardent, while the cupel is less red-that the undulations shall circulate in all directions, and that the middle of the metal shall appear smooth, surrounded with a small circle of litharge, which is being continually absorbed by the cupel. This treatment must be continued until the metal becomes bright and shining, or is said to 'lighten;' after which certain prismatic colors, or rainbow hues, suddenly flash across the globules, and undulate and cross each other, and the latter metal soon after appears very brilliant and clear, and at length becomes fixed and solid. This is called the 'brightening,' and shows that the separation is ended. In conducting this process, all the materials used must be accurately weighed, especially the weight of the alloy before cupellation, and the resulting button of pure metal. The difference gives the quantity of alloy."

When the operation is completed, the cupel is to be withdrawn from the fire and allowed to cool, and the metallic button then removed with the pincers. If the assay is a good one, it will detach easily. The button should be round and brilliant upon its upper surface, but rough and striated at the bottom. If its surface is dull and flat, too much heat has been employed; on the contrary, when it is spongy, adheres tenaciously to the cupel, and contains scales of litharge, there has been a deficiency of heat, and the fire must be again urged and the flowing of the metal promoted, by adding to the cupel a little powdered charcoal. Complete fusion is indispensable to the success of the operation. If too much lead has been added, the cupel is allowed to cool, the button carefully separated so as to be free from adherent particles of ash, and transferred to a fresh cupel and the process continued. In experienced hands, the pneumatic blast, p. 169, may be made to replace the furnace in the process of cupellation.

Cupellation in Taylor's Muffle.—Mr. T. Taylor (Memoirs of Chem. Soc., vol. iii. p. 316), claims for his new form of muffle the following advantages:—"1st. Crucibles may be maintained at a much higher temperature than can be readily obtained when the ordinary muffle is used, while the degree of heat and the quantity of air admitted may be regulated with the greatest nicety. 2d. Owing to the greater draught of air, the oxidation of the lead (in the process of cupellation) is more quickly effected; and lastly, by looking through an opening in the furnace cover, the operation may be watched from first to last.

"Two black lead crucibles of the same size are ground flat, so that when applied one to the other they may stand steady. An oblong or semicircular notch is cut out of the mouth of one of the crucibles, and a hole is also drilled through its bottom. This crucible, when placed on the top of the other, constitutes the muffle, and of course resembles in shape a skittle. To cupel with this apparatus, the lower crucible is nearly filled with clean sand, set upon the bars of the grate in the centre of the furnace and brought to a low red heat. The cupel containing the lead of the alloy is then placed upon the sand and immediately covered by the crucible, taking care that the notch in its side shall be opposite to, and correspond with, the furnace door; more fuel is added, during which it is well to cover the hole in the top of the muffle with a crucible lid in order to prevent the admission of dirt. When the muffle has become throughout of a bright red heat the furnace door is thrown open, and the ignited fuel gently moved aside so as to permit a view of the side opening in the muffle. The current of air which is thus established through the muffle instantly causes rapid oxidation of the lead, and this may be regulated at pleasure by closing the door more or less. If from the fuel falling down, any difficulty should be experienced in maintaining a free passage for the air, a portion of a porcelain tube, or a gun-barrel, may be passed through the furnace door to within an inch of the muffle; but this proceeding is generally rendered quite unnecessary, by taking care to place some large pieces of coke immediately round the door of the furnace.'

# CHAPTER XVII.

### SUBLIMATION. --- DISTILLATION.

WHEN simple or compound bodies which are either wholly or in part capable of assuming the aëriform state are subjected to heat, they or their most volatile constituents, upon reaching the required temperature, rise in the form of vapor. If these vapors, in their transit, are intercepted by a surface of a lower temperature, they condense and take a solid or liquid form, according to their nature. If the product is a solid, it is termed *sublimate*, and the process by which it is obtained is SUBLIMATION;—if it is liquid or gas, it takes the name of *distillate*, and the operation which yields it that of DISTILLATION.

Both of these processes are indispensably useful in chemistry, for they afford the facility of taking advantage of the unequal volatility of bodies for their separation.

As instances of sublimation, we have calomel and corrosive sublimate made by heating equivalent proportions of sulphate of mercury and common salt; benzoic acid evolved from the gum; pure indigo from the commercial article, and camphor from the crude material. Iodine is sublimed to free it from impurities; biniodide of mercury to convert it into crystals; naphthalin to free it from empyreumatic matter, and succinic acid to separate water.

In like manner, multitudes of instances of the importance of distillation in the everyday-processes of the chemist and the manufacturer might be adduced. It is employed in the separation and rectification of alcohol, the preparation of the ethers, of many mineral and vegetable acids, and of a very great number of other chemical products.

### SUBLIMATION.

The implements of sublimation are manifold, and vary in size and construction with the quantity of the substance to be heated, the nature, degree of volatility and the affinity of the subliming body for the oxygen of the atmosphere. There are certain rules to be observed in order to a successful execution of the process; but whatever the apparatus, its arrangement and management must be such that there shall be no diminution of the temperature of the vaporized matter until it reaches the recipient in which it is to be refrigerated and condensed.

The covers of flat subliming vessels and the recipients or condensing portions of those of other shapes, must invariably be out of and above the fire and exposed to the cooling influence of air. When the sublimed particles are very volatile, it will even be necessary to promote their condensation by covering the recipient with rags, which are to be kept constantly wet with cold water or some other refrigerant.

The usual mode of heating subliming vessels is by the sand bath, but for some substances requiring a very high temperature for their volatilization, direct fire is necessary, and this is applied with the lamp in small and nice operations, and in larger ones with the furnace.

In order to prevent explosion, the small opening in the top, at the centre of the refrigerant, must be only closed with a plug of raw cotton and should be freed from obstruction by occasional poking with a wire. When the escape of vapor through this hole is rapid, the heat is too high and must be diminished immediately.

After the completion of the operation, the apparatus must be left to cool before it is opened or the recipient removed.

The necessary breaking of close vessels for the removal of the contents, renders their use expensive; whenever, therefore, the nature of the substance will permit, an alembic with detached head should be preferred. Such vessels are more economical and easy of management, but generally require that their joints be made impermeable by luting.

Sublimation in Tubes.—Sublimation is very available in analyses for detecting the presence of minute quantities of volatile metals, acids, and other substances, the implement for the purpose being a small tube of such forms as is shown in Figs. 168, 174. After the introduction of the substance, previously powdered and fired, the tube is drawn out at its open end to a fine orifice and the lower part heated gradually over the flame of a spirit lamp (Fig. 174). The volatilized portion will be condensed upon the sides of the upper and cooler parts. By dividing the tube with a file, the sublimate

#### SUBLIMATION IN FLASKS.

can be exposed for microscopic examination or removed for

further assays under the The tubes for blow-pipe. sublimation may be from 4 to 8 inches in length and from an eighth to half an inch in diameter, according to the quantity of the matter to be sublimed and the required delicacy of the operation.

Berzelius uses a tube entirely open at the upper end

for those sublimations in which there are two volatile products, of which one is to be drawn off entirely in the form of gas by the absorption of oxygen from the atmosphere and recognized by its odor, and the other condensed in the upper part of the tube, as for example a mixture of sulphur and selenium.

Faraday gives the form of a tube apparatus (Fig. 175) for condensing heavy vapors or easily fusible substances, as naphthaline, iodine, &c. The bent tube b is of a diameter only large enough to allow its free passage over the subliming tube a. The upper part of the middle portion of the tube may be kept cool

by paper or cloth wrappers moistened with water in order to promote the condensation of the sublimed matter. The heat of the spirit lamp is sufficient for these small operations, and the apparatus, as adjusted, may be properly maintained by the upright clamp, Fig. 139.

Sublimation in Flasks .-- Florence or sweet oil flasks are well adapted to purposes of sublimation on account of their cheapness, uniformity of thickness, and power of resisting high heats. Having received their charge they are to be imbedded in a sand-bath to a depth above the level of the contents, and heat is to be applied gradually until the proper temperature is arrived at. The position of the flask should be inclined so that its neck may lead directly into the recipient, as shown in Fig. 176. In this manner considerable quantities of matter may be operated upon even at high temperatures, the glass bearing a red heat without injury.



Fig. 175.

## SUBLIMATION IN RETORTS, --- CRUCIBLES.

Another mode of arranging a flask for this process is to connect its neck in the manner of a hood, with a long bent tube leading into the refrigerant and recipient, as shown in Fig.



177. The inconvenience of this arrangement is the condensation of the gaseous matter in the tube, the obstruction from which may, without great care, cause the explosion of the flask.

Flat bottomed flasks of thin German glass are sometimes



used, but they are more expensive than oil flasks. Their position in the sandbath is upright and the flange around their necks acts as a support for an inverted globular flask which serves as a recipient. This arrangement, shown in Fig. 178, is admirable for the sublimation of substances, the volatile products of which are so aggregated as to form what are called *flowers*.

Sublimation in Retorts.—Glass retorts are more expensive and less convenient than flasks, except for the sublimation of very volatile matters. They are arranged as shown by Fig. 183, the beak, like the neck of the flask, leading into a widemouthed receiver. The alembic, Fig. 179, is frequently substituted for retorts and is more convenient, as its head being

detached from the body allows the more easy removal of the sublimed product.

Earthenware retorts with loose heads, Fig. 180, to be fastened by pins and lute, are employed for sublimations requiring high temperatures.

Sublimation in Crucibles.-The crucible for this purpose

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may be of clay, platinum or iron, according to the nature of the substance to be heated. It is first coated with a layer of refractory clay paste and when this is dry, placed over a furnace fire. An inverted crucible of the same size with a small hole in its top, is then placed over as a recipient of the



vaporized particles. The top crucible must be above and out of the fire. When the operation is finished, and the apparatus has cooled, the top may be removed and the crucible emptied of its contents.

Sublimation in Shallow Vessels.—In the treatment of substances which sublime at a low heat, a plate or capsule resting upon hot sand and surmounted by a glass funnel or a cone of glazed paper, as a condenser and recipient, answers every purpose, particularly in the sublimation of organic substances. The top of the funnel and cone should be drawn out to a small opening, and when the operation is finished the contents of the refrigerant may be removed by a feather.

When iron capsules are used, as is often necessary in sublimations requiring high heat, they should be lined with a thick dough of fire clay. Capsules with flat bottoms and of thick sheet iron are most appropriate. Their dimensions may be six inches in diameter and  $\frac{1}{2}$  to 1 inch in depth. The top

B must be of earthenware and detached. The arrangement is shown in Fig. 181. This implement requires a furnace heat. The cover should be tightly cemented with fire lute, and when



the whole has cooled, the cover may be taken off and the adherent mass of sublimate removed with a spatula. The small

#### HYDRO-SUBLIMATION.

cone c is to be kept over the hole in the cover to arrest any escaping vapors. When it is necessary to probe the cavity it may be temporarily removed with the tongs. A diaphragm of porous white paper will arrest the passage of any empyreumatic matter and pass the sublimate free from color.

Two very concave watch glasses, placed the one upon the other with their convex surfaces outward, make a very neat subliming apparatus for minute quantities of rare matter.

Fig. 182.



Ure's Apparatus.-This is a very convenient arrangement, Fig. 182, consisting of two metallic, glass or porcelain vessels. The lower one is the recipient of the matter to be sublimed, and the upper a, which is the larger, covers the former, and is to be filled with cold water to be replaced as fast as it evaporates. When the process is completed, the sublimed matter can be removed from the exterior of the cover.

Henry's Apparatus for Hydro-Sublimation.-This arrange-



ment, shown in Fig. 183, has been proved by experience to be practically useful. It is employed in manufacturing laboratories for the sublimation of calomel, but is equally applicable for other substances; and by lessening the dimensions of the several pieces, may be made very convenient for experimental purposes.

It consists of a large globular glass vessel a, with a long, straight neck, and two short, lateral tubulures of equal width.

For manufacturing purposes the globe must be of stone-ware, and of two or more gallons capacity. In either case it rests upon the ledge of a blue stone-ware cylinder h, containing sufficient water to close the neck of the globe which dips lightly into it. One of the tubulures receives the neck of the retort b, and the other that of the still, Fig. 13, which furnishes the steam, or, what is better, the conduit pipe of the generator, Fig. 10. The retort b, of earthen-ware, or iron, coated interiorly with fire clay, is for the evolution of the calomel or sublimate in vapors. It is wholly enclosed in the furnace, and its very short neck passes immediately from it into the globe a, so as to prevent the condensation of the sublimate in the neck and upper portion. The joints should be tightly luted. The success of the operation depends mainly upon a proper management of the fire and supply of aqueous vapor. The heat should be just sufficient to drive over the sublimate slowly, and the steam should be supplied in large excess, and simultaneously with the appearance of the vaporized solid. For this purpose the steam conduit must be fitted with a cock for the regulation of the flow of its contents of vapor. As soon as the sublimed molecules come in contact with the aqueous vapor, they are condensed in the globe a, and precipitate as powder (p. 84) into h.

By increasing the size of the globe a, threefold, diminish-

ing the orifice of its neck by means of a small glass tube traversing a perforated cork, and by omitting the tubulure on the other side, the steam may be dispensed with for the sublimations of volatile solids into flowers. The neck in this case must point upwards.

For experimental operations, a small earthen-ware retort and glass globe will answer every purpose. The steam can be supplied from the copper washing bottle, Fig. 185, by substituting for the spirting tube d, a flexible leaden pipe c, which is to be connected with the neck of the flask by a coupling



screw a. The gas or spirit-lamp will furnish ample heat for the generation of steam in this apparatus. Fig. 185.

Fig. 186.



#### DISTILLATION.

A process by which substances are heated for the separation of a volatile from a more fixed portion. The apparatus for the purpose consists of a close vessel in which the heating takes place, a refrigerant for the condensation of volatilized particles, and a recipient for the retention of the product; the two latter purposes being often, however, fulfilled by the same vessel. When this product condenses as a fluid, the process takes the name of *liquid distillation*, and if as a gas, of gaseous distillation. The subjection of a body to very high heat, for the purpose of decomposing it and receiving the generated products, is called dry or destructive distillation. In SUBLIMATION, which may be styled solid distillation, the volatilized matter is received and condensed in one vessel without the necessity of an intermediate refrigerant.

The process of distillation is one of the most indispensable in chemical investigation, as by its aid we can not only separate liquids of different volatility, but also collect new volatile products which may result from the decomposition of single or mixed substances. As instances of its valuable use, we can by its aid separate the essential oil and volatile constituents of plants and of other materials,—recover alcohol, ether, or any valuable volatile liquid, from solutions in which they

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are solvents,—refine a liquid from its fixed impurities,—free it from fixed matter which it may have in solution, and, aided by an absorbent material, remove any contained water. Moreover it allows the collection, either free or in solution, of gases generated by chemical reaction.

The Still.—The still is the common implement used in large operations of liquid distillation. It is generally made of copper, and is tinned internally. A convenient form has already been fully described at *page* 44. The figure below, Fig. 187, exhibits another of handsomer appearance, but constructed upon similar principles. It is mounted in brickwork,



but can as readily and with as good results, have its position in the more economical iron cylinder, Fig. 12.

By way of illustrating the necessary manipulations, we will describe the different steps of the operation as commonly performed.

The substance to be distilled is placed in the body A, the pewter or tinned copper head C, is luted on and adjusted to the pewter worm E, and the fire is lighted in the furnace. To insure facility of management, the several parts of the arrangement should be made so as to fit accurately to each other. As the heat increases, the contents of the body or cucurbit begin to boil, and that portion volatilizable at the temperature of the applied heat mounts in vapor to the head

or capital c, there partially condenses, runs into the beak or neck D, and ultimately into the spiral worm E, where, together with any uncondensed vapor, it is liquefied and cooled by the surrounding water previous to its exit into the recipient P. which may be an open pan if the product is not volatile, and a glass bottle or carboy if it is. The cooler I J K L, in which the worm is immersed, consists of a wooden cistern filled with water which requires to be constantly kept cool; for this purpose, therefore, there is a conduit N M attached, which receives cold water from the hydrant pipe R, and conveys it in a constant stream to the bottom of the cistern, so that it may displace the heated water, which has become lighter by expansion, through the lateral outlet at the top. This water, already heated, can be more economically used for making distilled water than when cold, as it takes less fire to boil it when transferred to the still. If the cistern is kept clean, it makes an excellent reservoir for the supply of hot water to the laboratory, as the still is frequently in use for making distilled water, and for other purposes.

When fresh additions of liquid are to be made they can be poured through the tubulure A. This saves the trouble of taking off the head of the apparatus, which need only be removed after the completion of the operation for the purpose of cleaning the still and its parts. As the residuum is in many instances as much the object of the process as the *distillate*, it must, when such is the case, be carefully removed from the still, and transferred to a suitable vessel for preservation or further reaction.

The size of the still varies with the amount of material to be operated upon. For the ordinary purposes of the laboratory it need not exceed fifteen gallons capacity. It must be proportioned so as to have as much heating surface as possible, while, at the same time, its height is sufficient for the foaming and frothing of its contents without danger of their boiling over into the neck.

We have advised a spiral worm because that is the usual form of refrigerants, an important point in the construction of which is to provide as much cooling surface, and consequently as great a length of pipe as possible in a small space. Schrader's condenser, Fig. 188, which is preferred by some manufacturers, because more easily cleaned, consists
of a metallic ball, the upper part of which projects above the

water contained in the cooler. From the lower side of this ball three straight tubes proceed, and conduct the vaporized particles downwards into the exit tube with which they connect. The exit tube is closed at its upper end with a cock, and open at the other for the escape of the condensed liquid.

Whatever the form of the refrigerant, its mode of action is the same. It is constructed so as to facilitate as much as possible the perfect and rapid condensation of the enclosed vapors. The greater the amount of surface which it presents to the water, the more effectual its action; for the sooner the heat absorbed by the distillate in assuming the gaseous form,

Fig. 188.

is abstracted by the surrounding water, the more rapidly it becomes condensed and cooled. The condensation may be hastened by surrounding the recipients with a frigorific mixture, which, if the vessel be of glass, must be at first applied gradually, lest its too sudden cooling causes its fracture.

Distillation in Retorts.—Retorts are egg-shaped vessels, answering a more convenient purpose than the still in the nicer distillatory operations. They are mostly of glass, but for some processes those of porcelain, earthen and stone-ware, platinum and iron, are necessary. Retorts are also used in technical operations, and the laboratory should be supplied with a series ranging from those of an half ounce up to several gallons capacity. Glass retorts should be made of hard, white glass, free from lead. Those of German crown-glass are very thin, but of uniform thickness and of sufficient strength, and moreover withstand both high temperatures and the corrosive action of acids and alkalies. The surface must be perfectly smooth and free from blur or striæ.

Some judgment is required in the selection of retorts. One properly constructed is exhibited in Fig. 189. It is seen that the neck proceeds laterally from the summit of the body, forming a wide tube at its origin which tapers gradually into a narrow beak. The arch of the retort should be so fashioned as to reverberate any particles of boiling matter that may spirt upwards against it, and thus prevent their overflow into the beak. A large neck facilitates the process, because it allows more room for the accumulation of vaporized matter, and presents a proportionally less surface to the cooling influence of the atmosphere.

It is very essential that the curve between the neck and the body a, Fig. 189, should be so formed as to make the *straight* line a b form an obtuse angle, with the dotted line b a. If, on the contrary, it is made as shown by Fig. 190, which presents the usual form of those sold in the shops, the





Fig. 191.

vapors, condensing in the arch as far as the dotted line a b, fall back again into the body, whilst, in Fig. 189, the dividing line, from which the distillate commences to flow, is much nearer to the body. So that a retort, like Fig. 189, will distil twice as rapidly as another similar to Fig. 190, which, however, when it has the form shown by the dotted lines c d a, becomes equally convenient.

The pear-shaped retort, Fig. 189, being deeper in the body or bulb, is better fitted for distilling volatile substances, and others which foam and swell upon being heated. The globular form, Fig. 191, presents less depth, but more heating sur-

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face, and is, therefore, better adapted to those liquids which boil quietly and distil more slowly.

A great improvement to the plain retort, is the addition of a glass stoppered tubulure to the neck, as at Fig. 191. The tubulure should have its position exactly as shown in the cut, so that the vapors condensing about it may flow back. Tubulated retorts are preferable, because they are more readily cleansed and charged than those of plain shape; moreover, they admit of fresh additions to their contents without the necessity of disturbing the arrangement.

If the distillation is to be urged over an Argand lamp, the neck of the receiver to be attached to the retort may be long, and the connection may be made by inserting the beak of the latter in its mouth, Figs. 192, 193, 194, and by rendering the



Fig. 194.



joint air-tight with a wrapper of India rubber cloth, as shown at R in the figure. The funnel D is charged with water which flows in a thin stream, regulated by the cock in the barrel, upon the receiver covered with sponge or bibulous rags and resting in a capsule c.

#### DISTILLATION IN RETORTS.

If the retort is to be heated in a furnace, and the receiver is globular, the junction of the beak and tubulure is tightened by means of a perforated cork through which the beak passes, and furthermore, if necessary, by a coating of flaxseed and whiting lute. The arrangement is shown in Fig. 195. The



receiver B resting upon a straw ring in a wooden pail, is cooled by a stream of water from the hydrant pipe I, which, as it becomes warm, flows off into the funnel d leading into the drain.

In order to increase the surface of the beak, and consequently to facilitate the liquefaction of the vapors passing through it into the receiver, there is often placed between the beak of the retort and the tubulure of the receivers, but connected with both, an adapter, Fig. 196, a pointed conical tube of white glass, free from lead, which, when leading from



a condenser to a bottle, takes a bent form as shown in Fig. 197. Fig. 198 exhibits a complete arrangement of a distillatory apparatus, combining a tubulated retort with an s tube, an adapter, a recipient placed in a vessel with a constant stream of cold water flowing into it, and a syphon tube with a second receiver. The curved adapter is needed where the receiver rests vertically instead of horizontally.



As it is requisite, frequently, in distilling volatile liquids, to have a larger extent of cooling surface than is presented by the globular receiver, another form has been devised by Liebig which is very convenient. It consists of a glass tube 25 to 30 inches long and one inch wide, connected with the



beak of the retort and running through a sheet brass cylinder of 20 inches in length and two or more inches diameter. The

#### DISTILLATION IN TUBES.

metal tube is closed at each end with perforated corks, through which the glass rod passes, and is held in a central position. A constant stream of cold water supplied through the funnel tube, passes into the cylinder, surrounds the glass tube, con-

Fig. 200.



denses the vapor therein contained, and becoming warm, passes out through the exit pipe to give place to cooler water. The figure above exhibits one mounted upon an iron stand with joint and sliding rod; from which, for small operations, it may be detached and supported by a wooden clamp.

For micro-chemical distillations, the necessary apparatus may be formed of glass tubes blown into proper shape over the blow-pipe table. Fig. 200 exhibits Clarke's tube retort and receiver:—a the retort of an ounce capacity, b the receiver 8



by three quarter inches, and c the distilled liquor. The junction of the retort and receiver (d) should be hermetical.

Plain bulbs a and tubulated b, Fig. 201, are other forms:—the tube b of the latter serves for the suction of the liquid to be heated, and may afterwards be sealed in the flame of a spirit lamp.

The means of heating these small vessels, are the small spirit lamps Figs.

118, 116, except when it is required to modify the heat by a sand-bath, which requires a larger lamp. The clamp supports p. 176, heretofore described, are of very great convenience in the adjustment of these tube arrangements.

A simpler form of tube retort is shown in Fig. 202. It is readily made by closing a tube at one end and bending it in a zigzag direction as represented in the drawing. The liquid to be distilled is at  $\alpha$  and the recipient at b. To render it



applicable to the generation and collection of gases, the tube may be drawn out at its open end and bent downwards if necessary to reach the receiver.

Platinum Retorts.—The size of these vessels varies from a quart down to an ounce, these capacities being adapted to all the purposes of an analytic and pharmaceutic laboratory. The usual form is shown in Fig. 203. The body a is nothing more than a stout crucible with a thick rim

d. The head b, with the helm c, should be hammered from one piece or else very closely welded together, and it should be ground at its rim so as to fit perfectly to the mouth of the still.

Platinum stills are very useful for destructive distillation, for determining the amount of matter, if any, lost by substances at a

red heat, for the distillation of matters not readily volatilized, of those which corrode glass, &c., and consequently, as a substitute for lead in the preparation of fluohydric acid.

Those of a large size should be fitted with handles so as to diminish their liability to defacement by transfer from place to place. When heated over charcoal, they should be well payed over with an external coating of fire clay paste. Otherwise, the directions for using the platinum still, are the same as those given for the crucible at p. 197. The gas or spirit lamp will furnish the amount of heat required for most operations.

Iron Retorts. — All iron retorts should be of cast metal. A very neat form for small operations is shown by Fig. 204. A simpler and more economical apparatus is a mercury flask, Fig. 205, with an iron gas tube or gun barrel screwed into the top, and reaching nearly to the bottom, and another tube bent down-





# 242 DISTILLATION :--- IRON AND PORCELAIN RETORTS.

wards. This arrangement, well fitted for distilling dry

Fig. 205.



substances which require a high heat, may be modified by

Fig. 206.

removing the centre pipe and inserting a screw plug, and thus be made well adapted to the distillation of mercury. For distilling naphtha, caoutchicine, and similar substances, the usual form of a glass retort is sometimes preferred. Fig.

206 exhibits one fitted with an iron tube or conduit, for connection with a condenser or receiver, which for mercury, may be an iron mortar or very thick glass bottle half filled with water.

Plate iron retorts, sometimes used for the generation of gases by high heat, are referred to in the distillation of volatile substances.

All of these iron\* retorts are heated in furnaces, that represented by Fig. 206 being placed horizontally.

When not in use, they should be greased to prevent oxidation, and should be kept stoppered.

*Porcelain Retorts.*—These implements, of shape similar to those of glass, are only used for dry distillations; but require, that fracture may be avoided, to be very carefully heated. Being opaque, they have not the advantage of glass, which allows the inspection of the contents of the vessel.

\* One per cent. of platinum, it is said, renders iron resistant to acid and corrosive liquids.

Earthenware and Stone Retorts.—The application of this ware to the purposes of distillation is very limited. To render it impermeable by gases, the retorts should be wet with a solution of borax, or else payed over with a coating of paste made from 9 parts of clay and 1 part of powdered borax, and then heated to fusion and gradually cooled.

General Rules for Distillation.—In the distillation of substances which require a high heat, the vessel may be placed over the naked fire. If it is a metallic still, the cylinder, Fig. 12, affords every convenience for heating by this method. Luhmé's reverberatory furnace, Fig. 89, is the proper heating implement for earthen and metallic retorts; and the spirit or gas lamp for those of glass and porcelain. When the nature of the process requires a modification of the heat, it can be accomplished by means of intermediate BATHS, which will furnish any temperature required up to the boiling point—of mercury.

Glass and porcelain retorts should, if possible, never be heated over the naked fire, because of their great liability to fracture. The impossibility of maintaining a uniform heat is a serious objection to this mode, for the ebullition, though rapid, is also unequal. When the above vessels are thus heated, the same directions are applicable to their management as to that of earthen or metallic retorts, though in the use of the latter less care is requisite. The proper position of the retort is in the centre of the furnace, Fig. 183, upon a crow-foot or support, Fig. 107, resting on the grate. The retort having been previously charged, its beak is then adapted to the receiver, and the joints closed by lute. A very small fire is then ignited and increased, after the retort has become warm, till it reaches to within a line or two of the level of the contained liquid. If the coals project beyond this point, the surface of the dry or upper part of the retort acquires a temperature so much higher than that of the substance which is being heated, that the difference may cause its fracture when particles are projected against it. A certain degree of heat in the upper part of the vessel is, however, necessary, so that the opposite condition-the condensation of vapor in it, may not occur; and where the retort is heated unequally, it is sometimes necessary to place over it the dome of the furnace. For the same reason, also, when a retort is heated over a spirit or gas lamp, or by any other way in which the upper portion is

exposed, that part should be covered with a dome. Fig. 207 exhibits such a one of earthenware for large retorts. A cone



of pasteboard, Fig. 208, will answer better for smaller vessels. The notch in the front allows its adaptation to the neck; but while adjusted so as to effectually protect the upper part of the retort from contact with air, it must be supported so that its weight, when great, shall not endanger the safety of the retort.

The addition of the fuel should be gradual, so that the fire may be only sufficient to gently boil the contained liquid. The coals should be first ignited to expel moisture; and when the operation is nearly completed, the fire must be skilfully managed. For greater safety, the glass retorts should always be coated exteriorly with a paste of refractory clay.

When the Argand or gas lamp is used as the means of heating, the retort need only be arranged upon a support, and brought over the flame, which is to be applied gradually at first, and slowly elevated until the glass has become heated throughout.

Fig. 195 exhibits a retort properly located in a SAND-BATH, this being the mode of heating retorts for the distillation of volatile liquids, such as ethers and the like. The advantage of the sand-bath over the naked fire for heating glass retorts, particularly those of large size, is that it imparts a more uniform degree of heat, and prevents the possibility of fracture from sudden changes of temperature. The sand should be *fine*, and the layer upon which the bottom of the retort rests about an inch or two deep according to the size of the retort. The sand surrounding the retort should only reach to the level of the contained liquid, and should be removed gradually as it evaporates. To prevent condensation in the top, the upper portion of the retort may sometimes be advantageously covered with a woolen cloth.

When the operation is performed with a view to separate two liquids which boil at different temperatures, the retort must be either set in a bath which does not exceed the temperature at which the more volatile liquid escapes; or else, when otherwise heated, the temperature must be regulated by a glass thermometer, Fig. 84, entering the retort through its tubulure, and adjusted by a perforated cork.

When the boiling points of different liquids are nearly equal, the density of one of them may sometimes be increased by the addition of some soluble matter, which, if both liquids are to be saved, can afterwards be readily removed.

Too sudden ebullition must, in all cases, be avoided, and the fire should be *gradually* increased, whether the heating vessel be of glass or metal. The only exceptions to this rule occur in the use of water or saline baths.

Distillation of Liquids.—The STILL is the most convenient implement for the distillation of large quantities of material. Retorts are more applicable to nicer operations.

The arrangement of a retort for the process of distillation is very analogous to that of a still. The body is the recipient of the matter to be heated, and is the portion to which heat is applied; the beak is the condenser, and the glass receiver, the recipient of the distillate. The exit nozzle fitting upon the end of the worm and leading into the recipient, should never project so far as to dip into the distillate. If a globular receiver is not at hand, an ordinary glass bottle for retort distillations, or a carboy for those in the still, are excellent substitutes, as it is very easy to make the connection by using a curved adapter, Fig. 197, and adjusting it to the mouth of the receiver, as in all other cases, through a perforated cork.

Sometimes the receivers themselves are drawn out at the neck into a tube which enters a flask, as at Fig. 209, or else the tubulure is fitted with a perforated cork for the passage of a tube which answers as well the purpose of a conduit. The flask which receives this tube is also fitted with a perforated cork through which passes another tube, c, Fig. 210, for the escape of uncondensed vapors.

# DISTILLATION OF LIQUIDS.

The refrigeration of the receiver is readily accomplished by either of the arrangements shown at Figs. 194, 195.



The uncondensed gases are allowed exit through a small glass tube adapted to the tubulure in the top of the receiver, and leading upwards under a hood, or else downwards into a bottle of some fluid which absorbs them, and thus prevents the contamination of the atmosphere.

It is of great importance that the vessels in use for distillation should always be free from foreign substances; and both the retort, still, adapter, and worm, immediately after each process, should be cleansed by repeated rinsings with water, so that they may be clean and ready for the next operation.

As the ebullition of certain liquids is attended with foaming and spirting, it is necessary to break the force of these sudden eruptions of vapor, by some mechanical means. This can be effected often by the addition of platinum scraps to corrosive liquids, and of fragments of glass to those which boil at low temperatures and are without action upon it. This precaution prevents damage to the vessel and allows the boiling to proceed tranquilly. The use of the water bath obviates the necessity of this preventive and is almost indispensable in the distillation of liquids holding in solution certain vegetable principles.

In distilling oil of vitriol in a glass retort, the deposition of sulphate of lead endangers the safety of the retort and the purity of the distillate by an explosive ebullition. To avoid this difficulty, Berzelius sets the retort one-third into the truncated cone of sheet iron, Fig. 211, strews sand around the edge of the cone, surrounds it with brick, and hangs a flat cone of sheet iron about a half inch above the retort. The retort is half filled with acid, and coals placed on the cone inside the bricks. Another method he

pursues is to precipitate the lead salt by dilution with water, to concentrate the acid in a platinum capsule, and, finally, to distil in a dome-topped furnace, a quiet distillation being promoted by the introduction of platinum wire into the retort.

If the retort is tubulated, there is no difficulty in charging it neatly, because its contents can be added

without danger of spilling through a wide barreled funnel; but when plain, it is necessary, in order to prevent the adherence of particles to the sides of the beak, to stand it on end, as shown in Fig. 212, and to fill it through the neck by means of a straight funnel tube with its barrel reaching to the bottom.

The matters, if solid, should always be bruised or triturated to powder and added portionwise. In this way the neck of the retort is kept clean.

The joints of retorts and glass distillatory vessels may be luted with strips of muslin

soaked in a solution of bone glue. Those of metallic vessels, with a dough of whiting and flaxseed meal, which, when dry, may be rendered still more impermeable, by a covering of muslin, prepared as above, with bone glue. When one vessel is adapted to the other by means of perforated corks, these latter should be payed over with wax or more economically with the flaxseed and whiting dough. If the distillate decomposes organic matter, the use of corks, flaxseed and similar means, must be avoided, and the joints made tight by using apparatus, the connecting parts of which are nicely adapted to each other.



Fig. 212.



All matters which readily generate very volatile products should be distilled over BATHS, of which those made of liquids are to be preferred. It is very easy to arrange the retort in a suitable vessel, by resting it upon a braided straw ring, Fig. 213, and steadying its neck in a clamp support. The beak



of the retort should also be elongated by attaching it to a Liebig condenser, Fig. 199, so as to extend the cooling surface. If the distillate is readily condensable, the receiver may be kept sufficiently cold by a bath of cold water, as shown in Figs. 194, 198; otherwise the use

of FRIGORIFIC MIXTURES becomes necessary. The mixture should entirely surround the recipient, and as it becomes warm or liquefies, new portions must be added. If ice or other solid is used, the syphon in position, as shown at b, Fig. 214, will keep the pail constantly free from the liquefied excess.

Fig. 214.



The proper arrangement of an apparatus with Liebig's condenser, is shown in Fig. 215.

In the distillation, particularly of essences and oils, the material, if it is ligneous, must be soaked, previous to its transfer to the still, in which it should be made to rest upon a cullendered diaphragm, Fig. 15, to prevent contact with the heated bottom of the vessel. A proper vehicle is then added and the operation proceeded with. The water, or other liquid, and volatile matter are vaporized, and the two becoming involved pass over together. When these two products differ in density, as is commonly the case, a

# DISTILLATION OF VOLATILE LIQUIDS.



very convenient means of separating them as they pass over, is afforded by the Florentine

receiver, shown in Fig. 216. A is the body of the recipient, and D its mouth through which the distillate enters. The tubulure B is for the reception of the beak (a curved glass tube), c. As soon as the condensed distillate reaches the receiver, the lighter body rises to the top, and there retains its position,

and when the contained amount of the two fluids reaches the level of the mouth of the beak, the one which is most dense runs off. The level being kept thus constant, the lower stratum of fluid is separated from the upper as fast as any distillate comes over, leaving the lighter liquid to be emptied from the receiver after the completion of the process.

If the heavier product is the object of the distillation, as is sometimes the case, then the form of the recipient may be with advantage varied, as shown in Fig. 217. The stop-cock in the barrel, allows its separation and discharge from the lighter body as soon as a sufficient quantity has subsided.

When volatile oils and some other bodies are obtained by the above process, the water which is generally employed as the vehicle, and which is separated as we have described, should be reserved, as being already more or less charged



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with volatile matter, it is economically used in redistilling the

Fig. 217.



same material, in case it should yield its product reluctantly. This repeated distillation of the distillate over the same material is termed *Cohobation* and is resorted to necessarily, in many instances, that the material may be entirely exhausted of its volatile matter.

Rectification is the redistillation of the distillate, either alone or with some absorbent material to free it from water, acid or other impurity.

DISTILLATION OF GASES.—The term "distillation" cannot in all instances be applied with entire precision to the processes concerned in the manufacture of gases. But as gaseous bodies when intended to be retained are usually prepared by modes precisely similar to those employed in liquid distillation, it has been thought pro-

per to introduce their consideration in this place.

The generation and distillation of gases are generally made simultaneous operations. As their elasticity is such as to prevent condensation by ordinary means, they are either collected in solution with water, or other fluid, or in their gaseous state in gasometers. The arrangement of the required apparatus, though bearing analogy to that for ordinary distillations, differs in many little but material points.

If the gas is readily evolved without the aid of heat, as in the case of hydrogen, carbonic acid, sulphuretted hydrogen, &c., the generator may be a simple flask A, Fig. 218. This flask is the recipient of the material from which the gas is to be eliminated. The funnel tube, reaching nearly to its bottom, is the inlet of the acid, or other reagent, by which the action is to be produced, and the bent tube is for the exit of the disengaged gas. An ordinary wide-mouth green glass bottle will answer the purpose excellently in most cases, as exhibited in Fig. 219, which also shows an attachment by a flexible India rubber joint of an angular tube, for passing the gas through liquids when the influence of its absorption is required—as in the precipitation of solutions in analysis. When the generator is to be connected with a combustion or DESIC-

### DISTILLATION OF GASES.

CATION tube, as at Fig. 148, the bent tube can be removed. In this instance, and, indeed, with better results in all cases,



the angular tube should be blown with a bulb in its centre for the reception of a plug of raw cotton, which intercepts the passage of liquid.

The perforations in the cork must be only large enough for the transit of the tubes, and the joints must be perfectly tight. To render the cork itself impermeable, scaling-wax should cover both of its surfaces. This arrangement of the tubes obviates all liability of explosion. If condensation takes place in the interior of the generating vessel, the resistance from the funnel tube being more feeble than that opposed by the water of the trough or receiving vessel, the air enters. So also, if from any cause the passage of the gas through the exit tube should be obstructed, its pressure upon the liquid in the generator forces it upwards through the funnel tube, so that it may escape instead of being allowed to accumulate until explosion takes place.

When it is desirable to have the gas free from impurity, an indispensable consideration when it is to be used in analyses, it should, previous to its entrance, be passed through a small quantity of water, or other fluid, which will dissolve out or chemically attract such foreign matter as might have an injurious effect upon the liquid to be acted upon. A suitable arrangement for such purposes, and one well adapted to the generation of sulphuretted hydrogen gas, is shown in Fig. 221; A is the bottle containing the protosulphuret of iron,



water, and sulphuric acid, and provided with funnel and disengagement tubes as usual, the latter plunging in the water of a smaller bottle B, from which a disengagement tube D nearly as high as that of bottle A issues, so as to lead the gas disengaged, into a beaker or other vessel containing the liquor to be operated upon: But the first disengagement tube c is in two pieces united by Indian rubber, and the bottles A B are connected together by a strong band of sulphurized Indian rubber, or of gutta-

percha G, so that the two bottles may be lifted at once as if they were in one, wedges of cork E E being forced between the two bottles so as to keep the strip of Indian rubber G and the tube C properly adjusted.

When heat is required to cause or to promote the elimination of a gas, the generating vessels may be either tubes, flasks, or retorts.

The facility with which glass tubes may be fashioned over the blow-pipe flame, into any desired shape, renders them particularly applicable to small operations. Fig. 222 exhibits a tube-apparatus for the distillation of hydrobromic acid. It



consists of a glass tube A B, to which is adapted a disengagement tube conveying the gas under the bell c, filled with mercury. The bromine is placed at A, and at B are small particles of moist phosphorus intermixed with bruised glass. Gentle heat being applied at A, the vaporized bromine reacts upon the phosphorus and water, producing phosphorous acid, and disengaging hydrobromic acid gas, which passes over into the bell-receiver and displaces the mercury.

A test tube, Fig. 116, fitted with a bent tube, serves very conveniently for generating small quantities of gas for analytic or even experimental purposes. Any other forms that may be needed will be suggested by the requirements of the process or the ingenuity of the operator, and can readily be fashioned after the instructions given upon GLASSBLOWING.

Another very convenient form of tube generator is that known as Marsh's Arsenic Apparatus, Fig. 223.

It consists of an elbowed tube of Bohemian glass, fitted with a stop-cock and jet, the whole to be supported by a suitable stand or pedestal. The length of the tube may be sixteen inches, and its width three-fourths of an inch.

The elbow being charged with some pieces of purified zinc, the liquid containing the suspected compound is acidulated with oil of vitriol and poured into the long leg. The arsenical combination becomes decomposed by the nascent hydrogen

generated by the action of the sulphuric acid upon the zinc and water, and makes its exit through the cock at the short arm, as arseniuretted hydrogen, which may be recognized when ignited by its bluish white flame, and the appearance of the deposit upon a porcelain plate held over it. The stopcock allows the facility of regulating or stopping off the supply of gas when required.

Next to the tubes, a Florence flask is the most economical vessel for conducting small operations. Fig. 224 exhibits one undergoing the process of being heated by a small spirit lamp. The tripod upon which the flask rests, is surrounded by a tin plate screen of a foot in height, to confine the heat of the lamp. The exit tube conveys the gas into the beaker glass c, which contains the solution to be saturated. Flasks of very thin glass, and made uniform throughout, are blown expressly for this purpose, as shown in Fig. 225. The exit tube is bent so that it may be used with bell glasses over a pneumatic trough, as in Figs. 222, 243.

Retorts are only convenient when large quantities of ma-

Fig. 223.



#### COLLECTION OF GASES.

terial are to be operated upon, and for most operations they should be made of hard glass free from lead. For those pro-



cesses which require high furnace temperatures a metallic retort is needed. Fig. 226 exhibits one of iron. It is fitted



with a very convenient coupling or gallons screw by which the neck may be connected with flexible lead or other exit pipes. The accompanying circular plate, in two pieces, serves both as a cover to the furnace and as a support for the neck of the retort to retain it in place. The cheaper mercury bottle supplies the place of this apparatus admirably in nearly all instances, and with the gun barrel attachment, as shown in Fig. 226, or other tube, is particularly useful

in the manufacture of oxygen.

Collection of Gases.—Gases are either collected in the aeriform state or else in solution. When generated extemporaneously, merely as precipitants of some solution, they are passed through the liquid contained in a beaker glass, as at Fig. 224, and Fig. 221, a tightly stretched paper cover being in general all that is required to confine the unabsorbed excess in the vessel.

If a liquid, as well as a gaseous product, is generated

# COLLECTION OF GASES.

simultaneously, then the arrangement of the apparatus may be, as shown in Fig. 227. The use of this may be well illustrated by a reference to the manufacture of nitrous oxide gas. The nitrate of ammonia, the material from which it is generated, is placed in the retort, the beak of which is adjusted, by means of a perforated cork, to the tubulure of a globular receiver. This receiver in its turn is connected by bent glass tubes, rendered flexible by a caoutchouc joint, with a Wolffe's bottle. The latter, deriving its name from that of the inventor, consists of a bottle, the size of which may vary with the extent of the operation, having in this instance three tubulures, each of which is fitted with a perforated cork for the

Fig. 227.



passage of glass tubes. The first tube a b, Fig. 229, conducts the gas from the receiver. The central tube c d, acts as a safety valve: the gas cannot escape through it, but if condensation ensues within the bottle, the external air rushes in and prevents the liquid from running over into the recipient or next bottle, if there are two connected, by reason of absorption. The third tube a, is the exit tube for conveying the gas directly into the recipient.

The retort, receiver, and Wolffe's bottle having been connected together, and the joints hermetically closed, heat is then gradually applied by means of the spirit Argand lamp. The eliminated gas passes over into the receiver, there deposits the aqueous vapor with which it is involved, and continues on to the Wolffe's bottle containing water, in its transit through which it parts with the rest of its aqueous vapor, and its other impurities, and ultimately reaches the exit tube a. If the gas is to be received into caoutchouc bags, Figs. 129, 130, and pages 216, 255, for inhalation or other purposes, the connection may be made directly, as seen in the figure, by means of a gallows screw, which allows an empty bag to replace another which is charged whenever desired. If the bags are intended as reservoirs for the preservation of the gas, their necks are fitted with gallows screw stop-cocks and connecting nipples. Those of small size, for the purpose of inhalation, are fitted with an ivory mouth-piece m, Fig. 227.

If the gas is to be conducted into a Pepy's gasometer, as at Fig. 234, or under a bell-glass over a pneumatic trough, as at Fig. 243; then instead of the stop-cock there must be a flexible bent tube of shape similar to that attached to the flask, Fig. 219, adapted to the third tubulure of the bottles.

Lead pipe of small bore may be used as a conduit when the generated gas is not corrosive. The gallows screw then becomes the proper mode of connection.

To favor the condensation of the aqueous impurity of the gas, the globular receiver should be kept cool during the distillation. So also the Wolffe's bottle must be surrounded by water, or a cooling mixture, when the gas is very volatile, otherwise the elevation of temperature, which generally occurs, may cause its dissipation.

When two or more gases are generated simultaneously, they may be separated by the presence in the receiver of an appropriate liquid, which is a solvent of one, but not of the other of them. Thus oxygen may be freed from carbonic acid by passing the mixture through a solution of caustic potassa, which absorbing the acid, becomes carbonated, and allows



the transit of the purified oxygen. This mode is adapted for this and other purposes in organic analysis, the requisite apparatus being a five bulbed white glass receiver of the form shown by Fig. 228. Its arrangement for the purpose is given in Fig. 103, a being the combustion-tube, in which the substance to be analyzed is introduced after its mixture with oxide of copper or chromate of lead, from which it obtains the oxygen necessary for its

combustion. The figure represents the tube in its position during the analysis, in the trough-shaped furnace of sheetiron, in which it is heated by being surrounded with ignited charcoal. By means of a perforated cork, the combustiontube is connected with the tube in which the water produced by the combustion is condensed. It is filled with chloride of calcium in order to absorb all the vapors from the carbonic acid, which passes through it into the apparatus m r p, through which it would be forced, were it not absorbed by the solution of caustic potassa contained in the lower bulbs. After the completion of the combustion, the carbonic acid which remains in the combustion-tube is extracted from it, by breaking off its pointed extremity and applying suction to the other end of the potassa apparatus at p, by which air is drawn through the whole apparatus, and the carbonic acid absorbed by its passage through the solution in the potassa bulbs. The weight of the water and the carbonic acid, is obtained by weighing the chloride of calcium tube and the potassa apparatus before and after the combustion. For this purpose each may be conveniently suspended to the supplementary pan, Fig. 62, of the analytic balance, p. 105.

In the instance we have been referring to, the Wolffe's bottle is used for the purpose of retaining watery vapor or other impurities in its contained fluid. Its most common employment, however, is when the gas itself is intended to be preserved in solution in water or other fluid. For this purpose, the number of Wolffe's bottles is often increased to three or more, which are connected together by tubes with flexible joints. In this manner, any gas that has remained unabsorbed by the liquid in the first bottle, is successively exposed to the dissolving influence of that in the others, until it becomes entirely liquefied. The Wolffe's bottles may, in many cases, be well replaced by wide-mouthed jars or bottles, with two or three perforations in their corks, and which may be made to answer all the purposes of the more expensive apparatus.

Fig. 229 represents an apparatus for the generation and absorption of gas; a being the heating vessel, the contents of which should fill only half its capacity, so that they may not upon too sudden reaction, run over into the recipient: b, the recipient either for the condensation of aqueous

# 258 ABSORPTION OF GASES ;--- WOLFFE'S BOTTLES.

vapor, or the abstraction of impurity by a contained vehicle, and the three Wolffe's bottles, half filled with water, the



receptacles of the eliminated gas. As the volume of the liquid in the Wolffe's bottles increases proportionally to the amount of gas received and dissolved, they should not be more than half filled at the commencement. So also the intermediate receiver should have but a very shallow depth of liquid, else it will take up gas as well as the impurities, and thus cause The water in the first bottle is the first to become a loss. saturated, and all the gas which is not absorbed passes over into the second and third bottles. This arrangement is that usually adapted for the distillation of acid, ammoniacal and other gases, which are condensed by solution. When necessary, any other liquid may be made to replace the water in the bottles. For example, aqua ammoniæ when it is desired to make a chloride or carbonate of that base, and lime milk for the solution of chlorine, &c. &c.

When the gas is to be generated by reaction of liquid upon a solid, the latter must be put into the flask before the stopper and tube are adjusted. The liquid can then be added through the s tube as often as it is required to continue the disengagement. If the gas is heavier than the solvent liquid, the disengagement-tube need dip but slightly beneath its level; and vice versa.\*

Safety Tubes.—When in the course of distillation, a momentary suspension of the heat or generating impulse causes a partial vacuum in the heating vessel, the liquid into which the disengagement tube dips is forced by the presence of the atmosphere into its bore, and ultimately into the vessel itself.

This entrance of liquid into the generating vessel may result also from its sudden cooling, by the entrance of cold water or by other means.

The results of this absorption are sometimes the fracture of the vessel, and more frequently irreparable injury to its con-



tents. To obviate these inconveniences, we use a safety tube, the usual forms of which are shown by Figs. 231, 232. The first, which is called an s tube from its similarity to that letter,

\* There are several points to be remembered in the generation and collection of gases.

1. All gases owe their existence as such to a certain elasticity, by reason of which they press upon the sides of the vessels in which they are enclosed.

2. The tension or elastic force of a gas is proportional to its quantity:—it augments with the temperature and decreases by refrigeration.

3. The atmosphere weighs upon all bodies, its pressure being usually equal to the weight of a column of water 34 feet high, or of a column of mercury 30 inches.

4. That liquids (in equilibrium) press equally in all directions.

These facts, therefore, render necessary the use of the safety tubes c c c, Fig. 229, and Welters, or the s tube, Figs. 230, 231, 232.

### ABSORPTION OF GASES ;---SAFETY TUBES.

is most used, but either of them may be employed in the following manner. If the generating vessel has only one aperture, its cork having been tightly fitted and rendered impermeable by coatings of sealing wax or other cement, on its upper and lower sides, is then to be perforated with two holes, one for the transit of the s tube, and the other for that of the exit tube. The tubes being tightly adjusted in the holes as shown in Fig. 230, and the cork fitted to the mouth of the generating flask, a quantity of water or sometimes of other liquids, as mercury, is poured into the s tube. When, during the process, the level is stationary in the bulb B, and in the part A of the tube, the apparatus is hermetically closed. If, however, a condensation of vapor takes place in the interior of the flask, the external pressure of the atmosphere weighs alike upon the liquid F of the trough, and that in the s tube; but as the latter offers the least resistance, the air first makes its contained fluid advance towards the flask, and then enters in bubbles into it, thus preventing the absorption of liquid from the trough or receiver.

If mercury is used, its relative density to water must be remembered, and the column of metal in the curve, should be as low as possible. As a column of mercury requires one of water nearly fourteen times its height for its support, it follows that if the former is too high, the gas passes back more rapidly into the recipient during condensation, from the disengagement tube than the air traverses the metallic mass in the s tube. Mercury is used when the Wolffe's series comprises a number of bottles; so that the opposing force of the contained liquid may not be sufficient to cause the disengagement of the gas through the s instead of the exit tube. When operating with a mercurial trough, the s tube, Fig. 231, without a bulb is used. The quantity of metal which it should receive must however be such that the pressure of the gas will meet with as strong a resistance from it as the liquid in the trough. When flasks are replaced by retorts, the latter should be tubulated, so that the safety tube may be adapted to its tubulure by means of a perforated cork.

If the retorts are necessarily plain, as in certain distillations by furnace heats, then the safety tube can be attached to the disengagement tube E Fig. 243, over the blow-pipe flame. The liquid is introduced at H I. This kind of tube

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known as Welters is very fragile, and requires careful management and handling.

Gasometers.—When the eliminated gas is to be preserved free from air, in large quantities for laboratory purposes or transportation, it is received in gasometers.

*Pepy's Gasometer.*—The most convenient gas holder is that known as Pepy's, Fig. 233. It consists of a japanned zinc hollow cylinder 16 by 12 inches, surmounted by a trough B of 9 by 12 inches, making the total height of the apparatus,

Fig. 233.

Fig. 234.



including that of the supports, three feet. Near the base of the cylinder is a lateral gulley placed obliquely and cut with a thread to receive a screw plug which closes it hermetically.

The tube D, supporting the centres of the vessels, and fitted with a cock, allows a communication between the reservoir and trough. A second tube s c i at the side, also fitted with a cock, passes from the upper cylinder into the lower, and descends, as shown by the dotted lines, to within a few lines of the bottom. The stem e is merely a support, and serves no other purposes. The coupling and stop-cock in the side near the top serve for connection with a jet, or for fitting on a bag or bladder. The glass gauge k graduated into cubic inches, is adjusted firmly and hermetically by means of sockets at the top and bottom of the cylinder. To prevent fracture it is embedded in a frame-work of the same metal as the gasholder.

The manner of using the apparatus is as follows:-close the mouth g, and fill the reservoir with water. For this purpose the water is poured into the trough b, and allowed to run down through the opened tubes d and c. The cock fbeing also opened, the confined air escapes as the water enters, and in proportion as the reservoir a is filled, the water rises in the tube k and hence the latter will indicate when it is full. As soon as this occurs and water runs through the pipe f, the cock of the latter must be closed, and the residual air allowed to escape through the still open tube d. If, upon gently shaking the vessel, no more bubbles appear, then all the cocks are to be closed and the apparatus mounted over a tub, of diameter some six inches or more, greater than that of the gasometer and the gullet q, is then opened. As the highest part of the edge of the inner aperature of this gullet is lower than the lowest part of the edge of the outer aperature, by a half inch or more, the water cannot escape unless the air simultaneously finds access, inwards, from above, by leak holes or otherwise. If, after the gush of a small portion when the plug is first removed, there should be any further leakage, it will be indicated by the gauge tube.

All being tight, the beak of the retort, or end of the conduit tube of the generating vessel, is introduced into the gullet, as shown at h, Fig. 234. The eliminated gas entering the receiver, displaces the water which escapes at g, and falls into the pail beneath. As soon as the vessel is full, which will be known by the examination of the gauge, or when a sufficient quantity has been collected, the disengaging tube or beak is withdrawn, and the gullet closed with the plug.

If it is desired to fill a gas bag from the reservoir, it must be fitted with an appropriate cock and nipple for connecting with the coupling cock f, and the pressure of the water with which the trough b has been partially filled, should be made to act upon the gas by opening the cock c. So also, when the gas is to be transvased into bell-glasses, these latter are filled with water to displace all air, inverted, and then brought directly over the opened tube d, as shown in Fig. 234. As the water enters from the trough through the tube c, gas escapes into the jar by the exit pipe d. When the vessel is filled, communication must be shut off by closing the cocks.

When a greater pressure is required, than can be given by the water contained in the trough, it can be obtained by means of a long-barreled funnel o, Fig. 236. When this is screwed by its threaded nipple p, into the socket s, Fig. 234, and filled with water, there is a pressure of nearly six feet.



By abridging the length of the barrel to q, the pressure is diminished to a little less than four feet. When two of these gasometers, filled the one with oxygen and the other with hydrogen, are united by means of a double jet, Fig. 235, connected by its branches a b with the cocks f of the reservoir, they form what is called the hydro-oxygen or COM-POUND BLOWPIPE.

Mercurial Gasometer.-When the eliminated gas is soluble in water or altered by contact with that liquid, it may with propriety be collected over mercury. For this purpose Pepy has contrived the arrangement shown in Fig. 237, which obviates the expense and inconvenience of filling the cistern with mercury. It is made of iron, and consists of a bell A A B B, which has a cock C at its summit, and which is immersed in a cylindrical iron cistern M N O P. This cistern is the reservoir for the mercury, but in order that as little as possible may be used, an iron core D E occupies its centre and allows an interval between it and the sides of the cistern only sufficient for the jar and mercury to make it tight. A glass tube a b cemented tightly to the top of this inner cylinder, traverses it and serves as a conduit of the gas into the bell. which is maintained in a vertical position by a movable elbow adjusted to the frame of the apparatus.

A cock c is adjusted to the tube a b, and puts it in communication with the eprouvette or small inverted bell F, placed upon a dish containing mercury.

In using this gasometer, the cavity M N O P is filled with

mercury, the cocks c and c are opened, and after the bell has been pressed down to its full extent in the metal, the cock cis closed.



The disengagement tube is then introduced under the mouth of the eprouvette and the generation of the gas proceeded with. Each bubble as it enters the bell elevates it proportionately; and when it has received a quantity equivalent to the volume of the capacity of the tube and of the eprouvette, the cock c is to be opened again and the bell depressed. The greater part of the gas which has entered and the air with which it is mixed, is thus forced to escape. A repetition of this manœuvre two or three times will insure the entire expulsion of the air; when the portions of gas given off can be collected and preserved for use. When the bell is full, the cock c is to be closed and the retort J removed.

To transvase any portion of the gas that may be wanted for use, it is only necessary to put the tubulure c in communication with the vessel in which it is to be received, by opening the cock and then to depress the bell in the mercury.

A scale graduated upon the side of the bell will allow an estimation of the volume expended.

Deville's Gasometer.—This apparatus, constructed upon the same principle as Marriot's vase, is most used in organic analysis.

Fig. 238 exhibits the arrangement. A A is a tubulated bottle filled with water; and

bottle intervalue with water, and a a a is the tube for disengaging the gas from the generator. Each bubble of gas displaces an equal volume of water, which is conducted off by means of the syphon b b, and the process should be continued until the expulsion of all the water, save just enough to fully close the orifices of the tube.

When the gas is to be disengaged for use, fill the funnel E with water and open the cock f. The pressure of the water descending into the flask forces the gas into the tube i i.

A flexible tube H I can then be adapted to the orifice of

the vessel into which the gas is to be introduced.

This gasometer is especially employed for holding oxygen to be passed over oxide of copper in tubes, after organic analyses, and to remove all carbonic acid that it may contain, it is passed through a flask containing an aqueous solution of caustic potassa.

The small bottle c contains the concentrated sulphuric acid and the tube u, potassa in one branch and chloride of calcium in the other.

If the oxygen is kept in this holder for too long a time, it becomes altered and contaminated with atmospheric air.

PNEUMATIC TROUGHS.—In most manipulations with gases, 18



#### PNEUMATIC TROUGHS.

particularly when they are required for immediate use or for temporary purposes, they are collected over the pneumatic trough. As the bell glasses\* into which they are to be received, must first be freed from contained air, it is necessary to immerse them in an appropriate liquid. Water and mercury are the two fluids almost universally employed, the first being used for all those gases which are not soluble in it, and for some which are only so in a slight degree, and the latter for those which are absorbed by water, and which exert no chemical action upon the mercury. Hence the distinctive terms, water and mercurial trough.

Water Trough.-A wooden pail, square or oval, with a

• Bell Glasses—Gas Jars or Receivers.—The arrangement of an experimental laboratory is incomplete without a series of bell glasses, varying in size from a gill upwards to one gallon. They should be of glass, preferably of white glass free from lead, should be made so as to combine strength and neatness, and should have a knob at the summit for convenience of handling. The rim of the mouth must be ground perfectly smooth and level, so that when resting upon an unground glass disk or an even bottomed plate, the joint may be tight. Fig. 239 exhibits a plain jar for ordinary uses; and Fig. 240 a similar implement



tubulated at its summit and closed with a glass stopper. This opening not only allows the facility of forming connections with other apparatus, but also that of filling it readily with liquid merely by depressing it while unstopped, vertically in the water trough. The air escapes through the tubulure in proportion to the rise of fluid in the receiver, which when full is to be stoppered and placed upon the shelf to receive the gas. Sometimes the tubulures are fitted with stop-

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little management can be converted into a most convenient water trough, Fig. 243. It should be of capacity sufficient



to allow the thorough immersion of bells of any size required for experiment. One of a foot in depth, sixteen inches in length, and ten inches in width, will be very suitable;—a vessel G of this size fulfilling almost all the requirements of an

cocks, as seen in Fig. 241, which add to the convenience of the jars in many

operations, and particularly when it is desired to pass a measured quantity of gas from the receiver into another vessel attached as heretofore described at p. 109. For this purpose the bell must also be graduated as seen in Fig. 242.

If the vessel into which the gas is to be received is a bladder instead of a glass bell, it must be fitted with a coupling cock, freed from air as much as possible by compression with the hands, then connected with the air-pump, Fig. 32, and p. 109, or syringe, Fig. 244, and completely exhausted by suction. The cock is then closed, the bladder detached from the syringe, and adapted by its coupling to the cock of the bell. Communication being opened the gas passes into the bladder upon the depression of the jar.

By grinding the surface of the ledge very accurately, and fitting the mouth of the bell with a ground glass disc, which by the intervention of a little grease may



form an air-tight joint, gases may be retained unaltered for a limited period.

experimental laboratory. Near to the top, in the interior, should be lateral flanges for the support of a sliding shelf a. This sliding shelf should have sufficient surface for the support of several bells or jars F at a time, and may extend over half of the trough. It is perforated with holes for the reception of the beak of the disengaging vessel as seen at b, Fig. 243.

A very convenient water bath for the collection of gases, may be formed of a deep earthenware dish, or other vessel in common use, by the addition of the bee-hive shelf, Fig. 245.



This shelf, generally made of porcelain, is a substitute for the shelf of a regular trough, and serves for the support of the bell glasses or other receivers. It may be two inches in diameter and one inch high, for a dish one foot wide and two inches deep, and proportionally larger for one of greater

dimensions. The disengagement tube enters the semicircular opening at the base and delivers the gas into the receiver by its curved end protruding through the hole in the centre. Part of a loaded wooden box, a metallic support, or other extemporaneous arrangement may in many cases be so adjusted to take the place of the common, or the bee-hive shelf —and to answer every useful purpose.

It is indispensable that the trough should be made thoroughly water tight and should be well hooped. For further protection it might be covered over with several coats of plumbago paint. Leakage being thus provided against the vessel may be used upon either the operating or centre table. The stop cock near the bottom allows the exit of the water when it has become dirty, acidified, or otherwise unfit for use.

The level of the contained water must always be half an inch or more above the top of the shelf. When the bell is to be charged, it is first completely immersed in the water so as to expel all contained air, then taken up by the knob, raised to a vertical position and carefully slid along with its mouth in the water to the shelf and placed immediately over the hole through which the disengagement-tube enters. As the gas bubbles up into the bell the water is displaced, and when it is filled, it can, if necessary, be drawn aside and replaced by another. So the process is continued until all the gas generated has been received.

As the first bubbles of gas eliminated are contaminated with air, they should be rejected; consequently the beak of the disengagement tube ought not to be brought under the bell receiver until the gas commences to pass over freely.

The Mercury Trough .- The high price of mercury renders necessary an economical construction of the trough in which it is kept. Fig. 246 exhibits one of convenient form. It consists of a well-japanned castiron trough, twelve inches long, seven inches wide, and two inches deep. The bottom, towards the side, is sunk throughout its length into a well two inches wide and one and three-quarter inches deep, and is expanded at its end into a circular cavity. This cavity allows of the immersion of the tubes



or bells in a moderate depth of mercury without the expense of filling the whole trough being incurred; and the circular end being larger than the other portion of the canal, allows the use of receivers of from two to three inches in diameter.

When this cavity is not in use and the mercury required to fill it is needed in the other part of the trough, it is closed with an exactly fitting iron plug which accompanies the vessel for this purpose.

The trough is placed upon the operating table and is manipulated with in the same manner as the water trough. For the convenience of supporting tubes in a vertical position, there is generally an accompanying clamp with sliding rod, which is attached to the side.

The small mercurial trough of porcelain, Fig. 247, very useful in organic analysis, contains usually from ten to twelve pounds and serves also very conveniently for experiments upon a small scale.

Fig. 248 exhibits a cylindrical glass trough which is used with tubes in organic analysis. It is of glass, fifteen and a half inches high, and is widened at the top. The drawing represents the introduction of ley into a graduated tube by means of the pipette a over a column of mercury. The tip of the pipette is curved so as to facilitate its entrance under the mouth of the tube. This plan is adopted sometimes in lieu of that given at p. 256, for the separation of two gases,

by presenting to both a third substance for which either of them has an affinity.



In the course of time the mercury of the trough becomes debased, by use, either with water, metals, or suspended matters. The water being specifically lighter, may very readily be removed by spreading sheets of bibulous paper on the surface of the mercury and renewing them as fast as they become imbued. The metals are separable by distillation, the mercury passing over pure into the recipient.

If the impurities are merely coarse suspended particles as of metallic oxides, straining, by compression with the hands through a chamois leather bag, will retain them, and even most amalgams, while the mercury passes through the pores entirely or almost pure.

Gases collected over air.—Although chlorine can for temporary purposes be collected over hot water in which it is not dissolved, that body as well as iodhydric and bromhydric acids and certain other gases which are soluble in water, or which attack mercury, are sometimes collected in receivers or bottles filled with air.

If the gas is lighter than air, the end of the disengagement tube should reach to the upper part of the inverted vessel. As the gas enters, the air is displaced and goes out below, and the jar is known to be full when the gas escapes also. The jar must then be slowly and carefully removed so that the vacuum left by the withdrawal of the tube, may be com-
pletely supplied by the gas simultaneously entering, and its mouth be closed with a plate of ground glass, cork, piece of caoutchouc, or other suitable means.

When the gas is heavier than air, as is the case with chlorine, the disengagement tube should enter to the bottom of the receiver, the mouth of which should be closely covered with a pasteboard disk. When the gas begins to escape at the mouth, the jar is full, and, after being closed with a ground glass plate or other stopper, carefully removed aside.

Transfer of Gases.—It is frequently necessary to transfer portions of gas from a large vessel to a smaller one for the purposes of experiment or MEASUREMENT.\* Having already given the mode of transferring from a gasometer we will now speak of transvasement over troughs.

If the jar or reservoir of gas is still upon the shelf of the pneumatic trough, the smaller vessel which is to receive a portion of its contents is to be entirely immersed in the fluid of the trough, and whilst full, conveyed bottom downwards to the shelf and there placed, so that it will project over the edge about a third of its diameter. The reservoir is then brought forward and the mouths of the two put in connection as shown in Fig. 250 by inclining the reservoir so that their edges may be in contact;—the gas then passes up in bubbles and by a little dexterity the rapidity of its flow can be easily regulated.

At pages 109, 133 and 134, we have already given directions for the transfer of gases into tubes and globular vessels.

Bladders are filled from the cocked receivers, Fig. 241, as directed at p. 267. Bladders are cleansed by ablution in weak potash lye, subsequent washings in fresh water and drying. The caoutchouc bags, mentioned at p. 216, are however preferable.

\* As the volume of a gas confined in tubes, or other vessels, over mercury or water varies according to the pressure of the surrounding atmosphere, it becomes necessary in experiments on gases to observe the barometric pressure, or the height of the mercurial column in the barometer, at the time the volume of the gas is observed. Every laboratory ought, therefore, to be provided with a barometer, which should either be a good syphon-barometer or a cistern-barometer, in which the mercury of the cistern may be brought to the same level before observing the height of the column. The latter is generally read off in a scale divided into inches, tenths, and hundredths of inches. As the height of the' mercurial column varies according to the temperature, a correction must be made for the temperature of the mercury in the barometer, which, for this purpose, is furnished with a thermometer to be observed at the same time.

## 272 CORRECTION FOR PRESSURE AND TEMPERATURE.

When the filled receiver is to be removed from the trough for further essays with the gas, it should be gently slid off



the shelf into a flat-bottomed plate containing just enough water or mercury to seal the mouth.

Correction for Pressure.—The pressure of the atmosphere at the level of the sea is equivalent to 15 pounds upon each square inch of the earth's surface, and is capable of supporting a column of water 32 feet high, and one of mercury 30 inches. The standard pressure, therefore, by which' the variations at different levels, and indeed at the same level, from some unknown causes, are estimated, is thirty inches.

"The following is the rule for calculating the volume which a gas should possess at one pressure from its known volume at another pressure. As the pressure to which the gas is to be reduced is to the observed pressure, or height of the barometer, so is the observed volume to the volume required. Thus, suppose a volume of gas has been observed at 120 cubic inches, when the barometer is standing at 28.8 inches, we find its real volume at the normal pressure, thus:

"As 30: 28.8: 120: 115.2 — the volume which the gas would occupy at 30 inches barometer; or, if the barometer at the time of the experiment stands at 30.6 then

"As 30: 30.6: 120: 122.4 = the volume which the gas would cccupy at 30 inches barometer. When the correction of a gas is to be made both for temperature and pressure, the reduction is first made for temperature, and the corrected volume is afterwards reduced according to the pressure."

#### DISTILLATION IN VACUO.

"Corrections for Temperature.—According to the recent experiments of Regnault, it appears that a volume of gas expands by heat  $\frac{1}{459}$ th of its bulk for each degree of Fahr., and calling the volume of a gas at 0° Fahr. unity, its volume at any higher temperature is found by the formula 1 +  $\frac{\text{temp. Fahr.}}{459}$  The determination of the volume of a gas at

one temperature from its known volume at another temperature may be attained by the following formula :---

"Let t be the temperature Fahr. at which the volume of gas is observed, t' the temperature to which it is to be reduced, xthe observed volume at t, and x' the volume at t' required.

Then 
$$x' = \frac{(459 + t') x}{459 + t}$$
.

"*Example.*—Suppose the balloon to be filled with a gas at the temperature of  $50^{\circ}$ , and that it has been previously ascertained that its content is exactly 50 cubic inches. The above formula enables us to calculate the real volume of the gas at the normal temperature, thus:

$$\frac{(459+60)\times 50}{459+50} = 50.982;$$

so that we actually have in the globe 50.982 cubic inches of gas, and our calculation for specific gravity must be made accordingly.

"Suppose, however, that the temperature of the gas is  $70^{\circ}$ , or  $10^{\circ}$  above the normal temperature, we have then less than 500 cubic inches present, for

$$\frac{(459+60)\times 50}{459+70} = 49.054;$$

and our calculation must be made accordingly."

DISTILLATION IN VACUO.—This kind of distillation is resorted to for the production or purification of many volatile matters which are alterable in the air or in aqueous vapor. Retorts drawn out at their beak into a very narrow opening, or glass tubes, fashioned over the blow-pipe flame to suit the process, are the vessels commonly employed. After the vessel is charged, heat is applied to the bulb portion, and as soon as it becomes filled with vapor and all air is expelled, the tube should be heated over a lamp. After the annealing of the closed end, it, being intended for the reception of the distillate, should be exposed to the influence of cold while the process is being conducted. If the retort is used, the receiver with which it is connected must be warmed over the sand bath, and in delicate experiments exhausted by means of the syringe, Fig. 244, or air-pump, Fig. 32.

Dry Distillation.—The distillation of a solid alone and without contact with liquid of any kind is called dry or destructive distillation. The process is resorted to for the decomposition of certain bodies more particularly organic, and their resolution into new compounds by an interchange of elements. The products are generally liquid and gaseous, consequently the arrangement of the apparatus must be with a view to the preservation of both, the means for which have been already mentioned. Dry distillation requires a high heat and consequently very refractory vessels which must be kept closed. As examples, citric acid, by this process may be converted into carbonic acid and oxide, acetone, aconitic acid, and water; fatty bodies modified into new substances and resins transformed into oily liquids and gaseous carbohydrogens.

In the dry distillation of nitrogenized bodies, the resultant products are complex, and contain nitrogen, ammonia, cyanogen, &c. For instance, indigo yields carbonate and prussiate of ammonia and kyanole.

In the arts, the dry distillation of wood furnishes charcoal, pyroligneous acid and pyroxylic spirit and creasote and tar; and that of bituminous coal and fat, illuminating gas.

### CHAPTER XVIII.

#### LUTES.

In all combinations of two or more pieces of apparatus or parts of them, there is a necessity, in chemical operations generally, of some means of hermetically closing the interstices of their joints so as to protect the enclosure from all outward communication. This is particularly requisite in SUBLIMATION, DISTILLATION and other heating operations, and indeed in all experiments with gases and liquids, wherever it is desired to confine the volatilized particles within the vessels and prevent their escape into the atmosphere and consequent loss.

To accomplish these ends we make use of lutes, which must vary in composition and mode of application with the material and construction of the apparatus, the temperature at which it is to be heated, and the nature of the generated products.

Caoutchouc.—This substance, in sheets, is particularly useful for forming flexible tubes by which joints may not only be rendered hermetical but also flexible. For delicate apparatus it is particularly applicable even at high temperatures. The tubes are made as directed at p. 216, and tied above and below the joint as at x, Fig. 166. Sometimes India rubber is replaced by muslin, payed over after its adjustment around the joints, with a paste made by the mixture of caoutchoue and spirits of turpentine.

Bladder.—Bladder well cleansed and divided into strips answers very well to a limited extent. For example, when moistened and coated with white of egg or solution of bone glue or of isinglass, it forms an excellent covering for the joints of retorts, tubes and the like, to the surfaces of which it adheres tenaciously. When, however, the contained ingredients generate corrosive vapors, and so rapidly as to strain the apparatus, the bladder is unserviceable.

Flaxseed Lute.—Flaxseed meal mixed to the consistence of a paste with water, milk, lime-water, or starch paste. This lute is very manageable and impermeable, but does not withstand a heat greater than about 500°.

If just the sufficient quantity of water be added to quicklime to reduce it to a dry powder, and that is mixed well and rapidly with white of egg diluted with its own volume of water, and the mixture spread immediately upon strips of linen and applied to the part, which is then sprinkled with quicklime, a good cement is made. Instead of white of egg, lime and cheese may be used, or lime with weak glue water or blood. This lute dries very rapidly, becoming very hard, and adhering strongly to the glass; but its great inconvenience is the want of flexibility.

In spirituous distillations, the joints of the apparatus may be closed very readily and effectually by a stiff paste of equal weights of whiting and flaxseed meal, mixed with water. We

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have found this lute invaluable notwithstanding its want of flexibility. It is the most easily made and most cleanly of all lutes, and when the pressure of the contained vapor is considerable, it can be covered with strips of bladder soaked in solution of bone glue.

Lime and Bone Glue.—Freshly slacked lime made into a thick paste with a strong solution of bone glue, makes an adhesive lute very applicable for closing the joints of vessels which are to be subjected to high heats, as, for example, those in which the distillation of lime and sal ammoniac for the production of gaseous ammonia is conducted.

Plaster of Paris.—Calcined gypsum made into a paste with glue or starch water, answers the same purposes as the above. When covered with strips of bladder it is rendered entirely impermeable by gases. A coating of oil or of a mixture of oil and wax has the same effect as the bladder, and the lute will then stand a dull red heat.

*Plastic lute*, is made by dissolving melted caoutchouc in hot linseed oil, adding finely powdered pipe clay and kneading the whole together into a homogeneous mass. The longer it is kneaded the better is its quality, and to prevent its hardening the caoutchouc should not be in deficient proportion. If it should become hard by keeping, it may be softened by kneading with a little spirits of turpentine.

This lute closes the joints without hardening, and can be removed at any time during the operation, to allow a change in the position of the parts of the apparatus.

For the distillation of acids or other corrosive vapors, it is very applicable.

Soft cement is prepared by fusing yellow wax with half its weight of crude turpentine and a little Venetian red, in order to color it. It is very flexible, and takes any desired form under the pressure of the fingers. It is extremely useful at common temperatures for tightening tubes in cork, and as a coating for rendering corks impermeable to gases.

Resinous, or hard cement, is made by fusing together at the lowest possible temperature, 1 part of yellow wax and 5 or 6 of resin, and then adding gradually, 1 part of red ochre, or finely powdered brickdust, (plaster of Paris succeeds very well,) and then raising the temperature to 212° at least, until no more froth arises, or agitation takes place, and stirring it continually until cold. This cement is employed in a hot state, and is very much used for fixing brass caps, &c., to air jars, and as an impermeable coating for the interior of wooden vessels.

Lute for joining Glass and Steel.—A saturated solution of mastic in alcohol, mixed with a solution of isinglass in dilute spirits, to which is added a small portion of galbanum or ammoniac, is an excellent cement for joining glass to glass or glass to steel. The mixture must be kept in a well-stopped bottle, and be always warmed previous to use.

Hover (Phila.) makes an excellent cement, which answers the purposes of the above, and is very useful in the laboratory for mending broken vessels for dry operations.

Lute for joining Crucibles.—A mixture of fine clay and ground bricks kneaded into a paste with water, holding in solution one-tenth of borax, answers admirably for luting the joints of superposed crucibles. An excellent lute for this purpose and also for metallic subliming vessels, is finely powdered Stourbridge clay, containing a little sal enixum, and made into a stiff paste with water.

Iron Cement.—This mixture is used for making permanent joints generally between surfaces of iron. Clean iron borings or turnings are to be slightly pounded so as to be broken but not pulverized; the result is to be sifted coarsely, mixed with powdered sal-ammoniac and sulphur, and enough water to moisten the whole slightly. The proportions are, 1 sulphur, 2 sal ammoniac, and 80 iron. No more should be mixed than can be used at one time.

Fire Lute.—The best fire lute is that employed by Mr. Parker, and is composed of good elay 2 parts, sharp washed sand 8 parts, horse-dung 1 part. These materials are to be intimately mixed; and afterwards, the whole is to be thoroughly tempered, like mortar. Mr. Watt's fire lute is an excellent one, but is more expensive. It is made of finely powdered Cornish (porcelain) clay, mixed to the consistence of thick paint, with a solution of borax, in the proportion of two ounces of borax to a pint of hot water.

Fat lute is prepared by mixing dry clay, in a fine powder, with drying oil, so that the mixture may form a ductile paste. It should be kept under cover, preferably in a greased bladder. When this paste is used, the part to which it is applied ought to be very clean and dry, otherwise it will not adhere. This lute is adhesive, and stands a pretty high heat, but requires to be fastened down with strips of bladder. Its greatest disadvantage is the difficulty of stopping holes which may be blown through it by escaping vapor.

Lead and Oil Lute.—Red lead mixed with boiled linseed oil is excellent for sealing the joints of steam-vessels. It hardens readily and bears a high heat.

Lute for coating Fire Vessels .- Faraday gives the following directions for luting iron, glass, or earthenware retorts, tubes, &c., for furnace operations. When the lute has to withstand a very high temperature, it should be made of the best Stourbridge clay which is to be made into a paste, varying in thickness according to the opinion of the operator. The paste should be beaten until it is perfectly ductile and uniform, and a portion should then be flattened out into a cake of the required thickness, and of such a size as shall be most manageable with the vessel to be coated. If the vessel be a retort or flask, it should be placed in the middle of the cake, and the edges of the latter raised on all sides, and gradually moulded and applied to the glass; if 'it be a tube, it should be laid on one edge of the plate, and then applied by rolling the tube forward. In all cases, the surface to be coated should be rubbed over with a piece of the lute dipped in water, for the purpose of slightly moistening and leaving a little of the earth upon it; if any part of the surface becomes dry before the lute is applied, it should be re-moistened. The lute should be pressed and rubbed down upon the glass, successively from the part where the contact was first made to the edges, until all air bubbles are excluded, and an intimate adhesion effected. When one cake of lute has been applied, and is not large enough to cover the whole required surface, another must be adapted in a similar manner. Great care must be taken in joining the edges, for which purpose it is better to make them thin by pressure, and also somewhat irregular in form, and if at all dry they should be moistened with a little soft lute. The general thickness may be about one-quarter to one-third of an inch.

Being thus luted, the vessels are afterwards to be placed in a warm situation, over the sand-bath or near the ash-pit, or in the sun's rays. They should not be allowed to dry rapidly or irregularly, and should be moved now and then to change their positions.

To prevent cracking during desiccation, and the consequent

separation of the coat from the vessel, some chemists recommend the introduction of fibrous substances into the lute, so as mechanically to increase the tenacity of its parts. Horsedung, chopped hay and straw, horse and cow-hair, and tow cut short are amongst the number. When they are used, they should be added in small quantity, and it is generally necessary to add more water than with simple lute, and employ more labor to ensure a uniform mixture. It is best to mix the chopped material with the clay before the water is put to it, and upon adding the latter, to effect the mixture, at first by stirring up the mass lightly with a pointed stick or fork; it will then be found easy by a little management, to obtain a good mixture without making it very moist.

The luting ought to be made as dry as possible, consistent with facility in working it. The more wet it is, the more liable to crack in drying, and *vice versâ*.

Mr. Willis recommends, when earthenware retorts, &c., are to be rendered impervious to air, the following coating. One ounce of borax is to be dissolved in half a pint of boiling water, and as much slacked lime added as will make a thin paste. This composition is to be spread over the vessel with a brush, and when dry, a coating of slacked lime and linseed oil is to be applied. This will dry sufficiently in a day or two, and is then fit for use.

Cement for Labels.—Gum tragacanth boiled with hot water makes the most adhesive paste for securing labels upon glass or other smooth surfaces. The addition of a few drops of oil of turpentine retards its decomposition and keeps it unaltered for a long time.

Mode of applying Lutes.—Lutes are applied whilst soft, being adjusted to the joints by the hand. As they become dry, occasional compression with the fingers is necessary to render them compact. So also when any leaks occur they must be closed with fresh portions of lute smeared over and pressed in by the end of the thumb. When bladder or muslin is to be pasted over lute, the joint made with the latter must first have dried.

When the operation is finished and the apparatus is to be disconnected, remove the lute first with the hands. If, as is often the case in the use of hard lutes in fire processes, they adhere tenaciously, then the use of a knife, or when the vessels are metallic, a chisel becomes necessary.

## CHAPTER XIX.

#### SOLUTION.

WHEN a substance added to a liquid is wholly or partially taken up by that liquid, it is said to be soluble therein. The liquid employed is termed the *solvent*, and its combination with the dissolved particles, a *solution*; and if the liquid has exerted its solvent power to the fullest extent, then the solution which it forms is said to be *saturated* because it can hold no more.

The variable degree of solubility in different liquids serves as a distinctive characteristic of bodies, particularly those which are solid.

Solution is either wholly mechanical, or else chemico-mechanical. In the first case it is a molecular division of a body, or, in other words, a diffusion of its particles in an appropriate liquid without any alteration of its original properties, save as to form and cohesion. Thus, for example, an aqueous solution of sugar or salt yields the whole of its charge by EVAPORATION, and one of sulphate of lime by the addition of alcohol, in which it is insoluble. Ethereal or spirituous solutions deposit their dissolved matter by DISTILLATION or CRYS-TALLIZATION; and some other kinds, that of gutta-percha, in chloroform, for instance, by PRECIPITATION with ether or alcohol. When the dissolved particles are thus recoverable again in an unaltered state, chemically considered, their solution may be styled *simple*.

In the second case chemico-mechanical solution, in contradistinction to that which is purely mechanical, is a process requiring the modification of a body by chemical action previous to its solution. Thus, for example, copper, iron, or any other base or acid, insoluble in the ordinary solvents, may be readily taken up by liquid acids or bases. But the liquid holds in solution a newly formed body entirely dissimilar to the original substance in properties, as appears when it is separated. In this, therefore, consists the difference between a simple or mechanical and a chemico-mechanical solution. As examples of this latter, iron may be dissolved in dilute sulphuric acid, but in the act is transformed into copperas: alkalies are taken up by acids, but become altered to salts; and oil in being dissolved by potassa solution is changed into soap. Hence it is that the chemical reaction is a preliminary step requisite to promote simple solution. The point of saturation in chemical solution is that at which the two bodies, invariably of opposite properties, have combined in proportions adequate to *neutralization*.

There are some exceptions to the above, which refer to certain instances in which acids and alkalies act as mere simple solvents, and without changing the original properties of the dissolved substance. For example, acetic acid dissolves certain phosphates and borates; aqua ammoniæ takes up carmine; potassa water the hydrated peroxide of tin, and hydrosulphuret of ammonia the deutosulphuret of iridium.

Solution is one of the most important processes in chemistry; it not only facilitates chemical reaction, but allows the separation of soluble from insoluble bodies, or parts of the same; and consequently the purification of the solution by subsequent FILTRATION, EVAPORATION, and CRYSTALLIZATION.

As regards the power of dissolving the greatest number of substances, water is the first in the rank of simple solvents, alcohol the next, and ether third. Then follow spirits of turpentine, pyroxylic spirit, the volatile and fixed oils, chloroform, and a host of other liquids suitable to particular substances. Of the alkalies, aqua ammoniæ or potassa are most used; the former preferably, because of its volatility and that of most of its salts. All of the common acids are employed, though some few only are of general application: such as the muriatic, nitric, sulphuric, acetic, and tartaric.

When the solubility of bodies is spoken of, it is in reference usually to water, that being the standard liquid. In testing the solubility of a substance, it is, therefore, usual to commence with that liquid, and if it fails, to proceed with the next in order; and always, for reasons below given, trying the experiment at varied temperatures, with gradually increased quantities if necessary.

A very convenient way of testing the solubility of a substance is by means of a test-tube. If solid, a small portion in powder is to be introduced and covered with distilled water,

#### SOLUTION ;-MEANS OF FACILITATING.

Fig. 251.



or the solvent to be used, and repeatedly agitated by the hand, the forefinger closing the mouth (Fig. 251) to prevent the escape of particles. If the matter is wholly soluble there will be no deposit at the bottom of the tube; if partially soluble the deposit will have decreased in bulk; if totally insoluble it will occupy the same space as at first. To determine as to the two latter results, a minute portion of the supernatant liquid is decanted and evaporated in a small platinum spoon or strip of window-glass over the spirit lamp, (Fig. 115;) if a residue remains it indicates that matter

has been taken up.

When heat is required, the lamp (as at Fig. 117) affords a convenient means of application. The procedure in such cases is the same as that above directed.

Volatile matters can in this way be recognized by their odor emitted at boiling temperature, or else by the taste which they impart to the liquid, or by some other characteristic test.

The solubility of a gas may be ascertained by passing up a given volume of water, or other fluid, the solvent power of which is to be determined, into a graduated tube filled with mercury and inverted, and then passing in similarly measured volumes of the gas. When absorption ceases, by bringing the interior and exterior levels nearly even, allowing for the small column of water, the remaining gas subtracted from the whole amount introduced will show how many volumes have been absorbed; knowing the relative sp. gr. of the gas and water, the volumes may be calculated to weights.

1. There are certain conditions which greatly facilitate the solution of substances:-1st, comminution, which increases the extent of surface; 2d, agitation, which promotes the frequent contact of all parts of the surface with fresh portions of solvents; 3d, the freedom from impurity of both the solvent and body to be dissolved. 4th, it is also influenced by the quantity and state of dilution of the solvent; 5th, by the temperature; 6th, by the mode in which the process is conducted.

2. Agitation is effected by stirring with glass rods when the containing vessel is open at the top. The rod should be rounded at the end over the blow-pipe flame, and to prevent

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its rolling from the table or top of the vessel upon which it should be placed, may be square instead of cylindrical as is usual. A very convenient and effective mode of bringing all portions of the liquid successively in contact with the substance to be dissolved, is to place the latter in a cullendered diaphragm suspended beneath the surface of the liquid. The first stratum of liquid in becoming saturated increases its density, and consequently descends and displaces a lower and fresher portion, which being in the same way surcharged in its turn, gives way to successive strata, and so the operation continues until the whole of the matter, or so much as can be, is taken up. This mode keeps the substance in constant contact with new portions of liquid, and is, in fact, a kind of *displacement* process.

When flasks or bottles are used the same effect may be produced by repeated shaking.

TRITURATION in a mortar and alternate decantation and fresh additions of the solvent greatly facilitate the solution of solid substances.

3. The purity of the solvent is an important consideration, for if it contain foreign matters they may impart a dissolving power which is not inherent in the pure liquid, or diminish that already possessed by it. Faraday makes the following excellent remarks upon this subject.

"It is necessary that the student be on his guard respecting certain variations in the solubility of bodies arising from the presence of other matters. He will continually find that, small portions of substances generally considered as insoluble. in water will remain in neutral solutions when some other substance is present, or because of slight mutual decomposition; and he will also frequently find that matter usually considered as readily soluble, is so with difficulty when in contact with substances with which it is not apparently in combination. Thus water boiled upon muriate of potash and phosphate of baryta will be found to contain more baryta than if boiled alone upon the phosphate; and on the contrary, if oxide of iron and alumina be precipitated together from a solution, it will be found much more difficult to dissolve the alumina by solution of potash than if it had been thrown down alone.

"The alkaline earths are remarkably soluble in solutions of sugar, and also, though to a less degree, in solutions of extract

and other vegetable matters: hence they are retained in solution at times in very unexpected situations, and might give rise to much uncertainty in the appearances and characters of other substances, unless the experimenter were aware of the general fact. Platina is not itself soluble in nitric acid, even when spongy and in its most comminuted form, but when alloyed in small quantities with metals dissolved by that acid, it becomes soluble with them, and in consequence appears now and then in situations where it is not expected. Tartaric acid or tartrates have an extraordinary power in rendering many metallic oxides soluble, which are not so by other acids without it; and still more in holding them in solution when such substances are added as in ordinary circumstances effect their separation. The oxides of bismuth, antimony, tin, and titanium, are easily dissolved by acids when tartaric acid is present; and being present, ammonia no longer has the power, upon its addition, of separating the oxides of iron, titanium, manganese, cerium, cobalt, nickel, lead, antimony, and the earths, alumina, magnesia, and yttria, from their solutions, and in certain cases even potash or soda fails so to do. Great advantage may be taken of this property occasionally, but sometimes it is equally disadvantageous in preventing the usual action of re-agents.\*"

4. In regard to the quantity and state of dilution of a solvent, it must be remembered that some substances require more of it than others for their solution, and that it should be in a greater degree of dilution. Therefore, in examining the solubility of a body always commence with small quantities, and increase both quantity and strength gradually as may be required.

5. Temperature exerts a considerable influence in the solution of bodies; and though in a few instances, as in the solution of lime, magnesia and anhydrous sulphate of soda in water, its elevation impairs the power of the solvent, yet as an almost universal rule it facilitates its action. The temperature must be adapted to the nature of the solvent and the substance to be dissolved, and of the solution formed.

It may be as well to mention that the caloric rendered latent at the moment of the liquefaction of a solid, which is being dissolved in a liquid, causes a decrease of temperature.

<sup>\*</sup> Annales de Chimie, xxiii. 356.

Solution in volatile liquids should be in most cases performed in the cold, and when of small quantities in narrow-necked flasks.\* If heat is required, especially when the vapors are inflammable, a retort or covered still must be used; and if the distillate is valuable, a recipient may be annexed to receive as much as comes over.

6. The mode of effecting solution varies with the substance under process: MACERATION, DECOCTION, INFUSION, DIGES-TION, BOILING AND DISPLACEMENT have each and all appropriate application.

In ordinary solution the solid should be added in portions, and sufficient interval allowed for the solution of those in the liquid before fresh are added. In case of foaming or effervescence an additional amount of fluid will produce a calm.

For solution in the cold or at slightly warm temperatures jars of hard German glass, Fig. 252, are very ap-

propriate vessels. The material in powder is added to the fluid in the jar and contact of fresh surfaces promoted by stirring with a glass rod. If the liquid solvent is volatile a glass stoppered bottle is a convenient substitute for the jar, agitation being effected by shaking it to and fro.

Some volatile substances which are insoluble in water under ordinary circumstances are taken up by it in the state of vapor. For this purpose both should be distilled together.

When solutions, emitting corrosive or disagreeable fumes, are being made in open vessels

the operation should be conducted under a hood, the barrel of which connects with the chimney-flue so as to ensure their exit.

The containing vessels should be those which resist the action of heat, acid, alkalies, and corrosive liquids.

For making saturated solutions of most substances, EBUL-LITION is necessary. For this purpose the solid must be boiled with the solvent until the latter on cooling deposits some of its charge. The cooled solution is then to be filtered.

\* When weighed quantities are to be transferred to a flask or other narrowmouthed vessel, the use of a funnel will prevent liability of loss. Any particles that may adhere to the side of the barrel can be washed down with portions of solvent.



Metals in their free state are dissolved one in the other by FUSION.

Solution of Liquids.—Agitation of the liquid to be dissolved together with the solvent generally effects solution. If upon repose there are two layers, then all the matter is not taken up, and that portion which represents the solution must be separated, and a fresh quantity of the solvent added. This process is to be repeated until all, or as much as possible, of the liquid is dissolved.

Solution of Gases.\*—The generation and solution of gases are generally simultaneous processes, and have been fully treated of at p. 258. When water is used it must be distilled and boiled to expel air. Viscid liquids are not less solvent than others, but take up the gas much more slowly. As a general rule the capacity of a liquid for a gas is proportional to its rarity. (Berzelius.)

The following table (from *Gray's Pharmacopæia*) of the solubility of some of the salts most in use will be found very convenient:—

\* Liquefaction and Solidification of Gases.—Faraday has succeeded (Ann. de Chim. et de Phys. 3d Series. Vol. 13, p. 121) in liquefying certain gases by the combined aid of pressure and refrigeration. Among them are olefiant gas, fluosilicic and hydrochloric acids. Alcohol was partially solidified in the same manner. Hydriodic, hydrobromic, and carbonic acids, sulphuretted hydrogen and ammonia assumed well defined solid forms.

The author thus speaks for himself:—"I sought in the first place to obtain a very low temperature, and employed for this purpose Thilorier's bath of solid carbonic acid and ether, placing it however under the recipient of an air-pump. By maintaining a constant vacuum, I lowered the temperature to such a degree, that the carbonic acid of the bath was not more volatile than water at the temperature of 86°, for the barometer of the air-pump stood at 28.2 inches, the external barometer being at 29.4.

"This arrangement made, I joined together, by means of corks and stop-cocks some small glass and copper tubes, so that with the aid of two pumps I was able to subject various gases to a pressure of 40 atmospheres, and at the same time to submit them to the intense cold obtained under the air-pump, and to examine the resulting effects. As I expected, the cold produced several results which pressure alone would never have done, and principally in the solidification of bodies ordinarily gaseous."

Name of Salt.	Solubility in 100 parts Water	Solubility in 100 parts Alcohol
	at 600 at Boiling point.	at 60° at Boiling point.
ALUMINA.		- 10
Acetate of	Undetermined	-
Arseniate of	Insoluble	
Borate of	Uncrystallizable	
Camphorate of	0.05	
Lactate of	Uncrystallizable	
Muriate of	Very soluble	100 at 54 <sup>1</sup> / <sub>2</sub> °
Nitrate of	Very soluble	100
Oxalate of	Uncrystallizable	2.91
Phosphate of	Insoluble	
Seleniate of	Insoluble	
Sulphate of	50	
Sulphate of, and Potash .	5.4 133.33	
Sulphate of, and Soda .	100	
Sulphite of	Insoluble	
Tartrate of	Uncrystallizable	2-91
Tartrate of, and Potash .	Uncrystallizable	
Tungstate of	Insoluble	
Urate and Lithate of .	Insoluble	
AMMONIA.		
Acetate of	Very soluble	Readily soluble
Arseniate of	Soluble	
Binarseniate of	Soluble	
Popposto of	Uncrystallizable	
Beletate of	Soluble	
Borate of	20	0 416
Camphorate of	1 22	0.410
Carbonate of (Seconi)	1 33 (Ilma)	
	20 (Brande)	
Chlorate of	Very soluble	
Chromate of	Very soluble	
Citrate of	Difficultly crystallizable	
Ferrocyanide of	Very soluble	
Formate of	Soluble	
Hydriodate of (or Iodide)	Very soluble	
Hydrocyanate of	Soluble	
Hydrosulphuret of	Very delignescent	
Hypophosphite of	Soluble and deliguescent	
Hyposulphite of	Very soluble	-
Iodate of	Sparingly soluble	
Lactate of	Uncrystallizable	
Meconate of	66	
Molybdate of	Soluble	

Name of Salt.	Solubility in 100 parts Water	Solubility in 100 parts Alcohol
	at 60° at Boiling point.	at 60° at Boiling point.
AMMONIA.		
		7
Muriate of (or Chloride of Ammonium)	36 100	4 75 do
Ammonium )		1.5 do. 2.5 .834
Nitrate of	50 100	19.16
Oxalate of	4.5 40.84	
Phosphate of	25 (Brande)	10
Bipnosphate of	Less soluble	
Purpurate of	.0066 much more	
Pyrolithate of	Soluble	
Suberate of	Very soluble	
Succinate of	Very soluble	
Sulphate of	50 (Brande) 100	
Tartrate of	60.03 304.7	9.91
Tungstate of	Soluble	2.01
ANTIMONY.		
Acetate of	Soluble (Ure)	1
Benzoate of	Soluble (Ure)	
Tartrate of	Very soluble (Brande)	
Potassio-tartrate of	7 50	
BISMUTH.		
	C.J. L.	
Acetate of	Insoluble	
Benzoate of	Soluble	Sparingly
Carbonate of	Insoluble	
Chloride of	Deliquescent	
Nitrate of	Decomposed	
Phosphate of	Decomposed	
Suprate of	Decomposed	
BARYTA.	5 at 50° 10 at 212°	
Acetate of	88 96	
Antimoniate of	Insoluble	
Antimonite of	Slightly	
Arseniate of	Diffeenter	
Benzoate of	Soluble	
Borate of	Very sparingly	
Camphorate of	Very sparingly	
Carbonate of	Very nearly insoluble	
Chiorate of	20 Voru sparingly	
Citrate of	Difficultly soluble	
Ferrocyanuret of	.0005 .01	
Hydriodate of (or Iodide)	Vory soluble	
of Barium)	very soluble	1

Name of Salt.	Solubility in 100 parts Water	Solubility in 100 parts Alcohol
	at 60° at Boiling point.	at 60° at Boiling point.
BARYTA.	5 at 50° 10 at 212°	•
Hydrosulphuret of Hypophosphite of Iodate of Lactate of Lithate of	11 50 Very soluble .33 1.6 Soluble Insoluble	
Muriate of (or Chloride of Barium) (Anhydrous)	36.8 68.5	$ \begin{cases} 1 \text{ at } 80^{\circ} & \cdot \\ 0.29 & \cdot \\ 0.18 & \cdot \\ 0.09 & \cdot \\ 0.09 & \cdot \\ 0 \text{ is } \end{cases} \overset{\text{ff}}{\underset{\text{f}}{\overset{\text{f}}}{\overset{\text{f}}{\overset{\text{f}}{\overset{\text{f}}{\overset{\text{f}}}{\overset{\text{f}}{\overset{\text{f}}{\overset{\text{f}}{\overset{\text{f}}{\overset{\text{f}}}{\overset{\text{f}}{\overset{\text{f}}{\overset{\text{f}}}{\overset{\text{f}}{\overset{\text{f}}{\overset{\text{f}}}{\overset{\text{f}}{\overset{\text{f}}{\overset{\text{f}}}{\overset{\text{f}}{\overset{\text{f}}}{\overset{\text{f}}{\overset{\text{f}}{\overset{\text{f}}}{\overset{\text{f}}{\overset{\text{f}}}{\overset{\text{f}}}{\overset{\text{f}}{\overset{\text{f}}}{\overset{\text{f}}}{\overset{f}}}{\overset{f}}{\overset{f}}{\overset{f}}}{\overset{f}}{\overset{f}}{\overset{f}}}{\overset{f}}{\overset{f}}{\overset{f}}{\overset{f}}}{\overset{f}}{\overset{f}}{\overset{f}}}{\overset{f}}{\overset{f}}}{\overset{f}}{\overset{f}}{\overset{f}}{\overset{f}}}{\overset{f}}{\overset{f}}{\overset{f}}}{\overset{f}}{\overset{f}}}{\overset{f}}{\overset{f}}}{\overset{f}}{\overset{f}}{\overset{f}}}{\overset{f}}{\overset{f}}{\overset{f}}{\overset{f}}{\overset{f}}}{\overset{f}}{\overset{f}}{\overset{f}}{\overset{f}}{\overset{f}}}{\overset{f}}{\overset{f}}{\overset{f}}{\overset{f}}}{\overset{f}}{\overset{f}}}{\overset{f}}{\overset{f}}}}}}}}$
Muriate of (or Chloride of Barium) Cryst }	43 (Brande) 78	$ \begin{pmatrix} 1.56 \text{ at } 80^{\circ} \\ 0.43 & . & . \\ 0.32 & . & . \\ 0.06 & . & . \\ 0.25 & . & . \\ y \end{pmatrix} \overset{\text{gd}}{\underset{\text{b}}{\overset{\text{gd}}{\overrightarrow{y_1}}}} .900 \\ .848 \\ .834 \\ .$
Nitrate of .   Oxalate of .   Phosphate of .   Pyrocitrate of .   Sulphate of .   Sulphate of .   Tartrate of .	35.18 at 58.9° 35.18 at 214.97° Nearly insoluble Insoluble 0.25 .066 .02 Insoluble Insoluble Slightly	
COBALT. Acetate of Antimoniate of Borate of Borate of Carbonate of Lactate of	Soluble Soluble Insoluble Scarcely Insoluble .026 (Ure) Very soluble Soluble Insoluble Soluble Soluble	100 at 54‡° Insoluble
COPPER. Acetate of Antimoniate of Benzoate of Borate of Carbonate of Chlorate of Chlorate of Chromate Citrate of Ferrocyanide of	(Ure) 20 Insoluble Slightly Insoluble Insoluble Insoluble Insoluble Insoluble Insoluble Soluble Soluble	

Name of Salt.	Solubility in 100 parts Water		Solubility in 100 parts Alcohol		
6	a1 600	at Boiling poi	nt.	at 600	at Boiling point.
COPPER.			_		14
Formate of	12				
Hyposulphite of	Soluble				
Muriate, or Chloride of .	Soluble		•	100 a	it 176°
Dichloride of	Nearly	insoluble			
Ovalate of	Soluble	2			
and Ammonia	Soluble	2			
and Potassa .	Soluble	3			
and Soda .	Insolub	le			
Phosphate of	Insolub	le			
Subnitrate of	Insolub	le	~		
Sulphate of	25	5	0		
Disulphate of	Insolub		1.5		
Sulphite of Protovide	Insolub		1		
Sulphate of and Potassa	Soluble				
and Ammonia	Soluble				
Ammonio Subsulphate .	66.6		-		
Tartrate of	Soluble				
Bitartrate of	Less so	luble			
Tartrate of and Potassa .	Soluble		-		1000
GOLD.					
Perchloride of	Soluble				
Protochloride of	Soluble				and the second second
IRON.					
Apotato (Brot)	Soluble				
Acetate (Per )	Uncryst	allizable			
Antimoniate of	Insolub	e			
Arseniate of (Prot.)	Insolub	le			
Arseniate of (Per.)	Insolub	le			
Benzoate of	Insolub	e			
Borate of	Insolub	le			
Citrate (Proto.)	Soluble	la soluble			
Citrate (Bi proto.)	(Verveo	uble and uncrys	->		
Citrate (Per.)	alliz:	able	~{		
Ferrocyanide (Prussian Blue)	Insolub	le	-		
Fluoride of	Insolub	le			
Gallate of Peroxide of .	Insolub	le			
Hyposulphite of	Soluble				
Molybdate of Protox of	Insolub	le			
Protochloride of	Soluble				
Perchloride of	Very so	luble .		100 a	t 176°
Nitrate of Protoxide of .	Uncryst	allizable	-		1.00
Nitrate of Peroxide of .	Very so	luble	1		
Oxalate of Protoxide of .	Soluble		1		
Oxalate of Peroxide of .	Scarcel	Y	1		
Prosphate of	Insolub	e	-		

Name of Salt.	Solubility in 100 parts Water	Solubility in 100 parts Alcohol
	at 60° at Boiling point.	at 60° at Boiling point.
IRON.	1	
Phosphate of Peroxide of Superphosphate of Succinate of Peroxide of Sulphate of (Cryst.) . Sulphate of (dry) . Persulphate of and Potassa Persulphate of and Potassa Persulphate of and Am- monia . Tartrate (Perolo.) of . Tartrate (of and Potassa .	Nearly insoluble Nearly insoluble Insoluble 76.238 (Brande) 333.3 Uncrystallizable Uncrystallizable Soluble Soluble 0.25 (Dumas) Soluble Uncrystallizable	Soluble Soluble
LEAD.		
Acetate (Cryst.) Acetate (Anhyd.) . Diacetate of Antimoniate of Benzoate of Borate of Borate of Carbonate of Chlorate of Chloride of Chloride of Chloride of Chloride of Chromate of Chloride of Ferrocyanuret of . Gallate of Hyposulphite of Superlactate of . Malate of . Molybdate of .	27 (Bostock) 29 Soluble Insoluble Insoluble Insoluble Insoluble Insoluble Soluble 3.33 (Brande) 4.5 Insoluble Insoluble Insoluble Soluble Soluble Soluble Soluble Soluble Scarcely Insoluble	12.5 (Brande) Soluble
Nutrate of .   Dinitrate of .   Oxalate of .   Phosphate of .   Succinate of .   Sulphate of .   Sulphate of .   Tannate of .   Tartrate of .   and Potassa .	13 {Scarcely at 60°, but much more so at 212° Insoluble Insoluble Insoluble Not absolutely insoluble Insoluble Almost insoluble Insoluble (Berzelius)	

Name of Salt.	Solubility in 100 parts Water	Solubility in 100 parts Alcohol
	at 600 at Boiling point.	at 60° at Boiling point.
LIME.	(Kirwan)	
Acetate of	Soluble	$ \begin{cases} 2.4 \text{ at } 80^{\circ} \\ 4.12 \\ 4.75 \\ 4.88 \\ 4.88 \\ \vdots \\ \frac{1}{2} $
Antimoniate of	Insoluble	
Arsonite of	Difficultly soluble	
Benzoate of	Sparingly soluble	
Borate of	Very difficultly	
Carbonate of (Anhyd)	Insoluble	
Chlorate of	Very soluble	Soluble
Chromate of	Soluble	·
Citrate of	Nearly insoluble	
Fluoride	Insoluble (Salubility and a seal	
Hypophosphite of	Solubility nearly equal	
Hyposulphate of	40.65 (Brande) 150	
Hyposulphite of	Very soluble	
Iodate of	20 100	
Iodide of Calcium	Deliquescent	
Malate of	.66 1.53	
Molybdate of . • .	Insoluble	
Muriate (or Chloride of)	400 at 60°	
Calcium) }	almost any quantity at	
Nitrate of	2200	161.66
Oxalate of	Insoluble	
Phosphate of	Insoluble	
Biphosphate of	Soluble	
Subphosphate of	Almost insoluble	
Succinate of	Difficultly soluble	
Sulphate of	0.301 at 50°	>- · ·
suprice of	(Nearly insoluble at 60°	
Tartrate of	but .16 at 212°	
Tungstate of	Insoluble	
LITHIA.		
Acetate of	Deliquescent	
Bicarbonate of	Slightly soluble	
Borate of	Soluble	-
Carbonate of	1	Insoluble
Chromoto of Lithium .	Very deliquescent	
Citrate of	Very difficultly soluble	
Nitrate of	Very deliquescent	
Oxalate of	Very deliquescent	
Binoxalate of	Less soluble	
Phosphate of	Insoluble	
Sulphate of	Soluble	1

Name of Solt	Solubility in 100 parts Water	Solubility in 100 parts
Name of Sait.	at 600 at Boiling point	at 602 at Boiling point
	at boining point.	at boining point.
LITHIA.	2	
Tartrate of	Easily soluble	
and Potassa	Easily soluble	
and Soda .	Easily soluble	
MAGNESIA.		
Acetate of	Very soluble	
Arseniate of	Deliquescent	
Arsenite of	Difficultly soluble	
Benzoate of	Soluble	
Borate of	Insoluble	
Carbonate of	Very slightly	
Chlorate of	very soluble	
		<b>(</b> 50 547
Chloride of Magnesium .	200 (Brande)	50 at 80° (Sp. gr.) .817
5		121 25 ) Sots   900
Chromato of	Very soluble	(ar as the copies ) isos
Citrate of	Difficultly soluble	1
Indide of Magnesium	Soluble	
Malate of	3.56 (Brande)	1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1
Malate of	6.66 835	
monybuate of	0.00	(Nearly insoluble in
Nitrate of	100	pure alcohol
Ovalate of	Nearly insoluble	C. P. B. O.
Phosphate of	6.66	
and Ammonia	Sparingly soluble	
Succinate of	Uncrystallizable	1
Sulphate of (dry) .	33.192 73.57	
Sulphate of (cryst.)	68.042 150.71	1 at 80° (Kirwan)
and Ammonia	Soluble	
and Potassa .	Soluble	
and Soda .	33.3	
Sulphite of	5	-
and Ammonia	Difficultly soluble	-
Tartrate of	Insoluble	
Tungstate of	Soluble	
MANGANESE.		
Acetate of	3	Soluble
Ammonio-chloride of .	Soluble	
Ammonio-sulphate of	Soluble	
Antimoniate of	Moderately soluble	-
Arseniate of	Insoluble Delianase (Burnel)	
Benzoate of	Incoluble (Brande)	-
Carbonate of	Insoluble	
Chromate of	Voru soluble	Seluble
Oralate of	Insoluble	Soluble
Dhaanhata of	Noarly insoluble	
Succinate of	1 (Ire)	
Succillate of a a	1 (()(e)	

Name of Salt.	Solubility in 100 parts Water	Solubility in 100 parts Alcohol
	at 600 at Boiling point.	at 60° at Boiling point.
MANGANESE. Sulphate of Hyposulphate of Sulphite of	31 (Ure) 50 (Brande) Deliquescent Insoluble	
Tungstate of		
MERCURY. Acetate of (Prot.) Acetate of (Prot.) Arseniate of Benzoate of Borate of Bichloride of Chloride of Chromate of Citrate of Fluoride of Nitrate of (Prot.) Nitrate of (Prot.) Nitrate of (Prot.) Sulphate of (Prot.) Sulphate of (Sub.) Tartrate of	0.16 (Braconnot) Readily soluble Insoluble Insoluble 6.25 (Brande) 33.3 .00833 at 212° (Dumas) Insoluble Insoluble 54 Soluble and decomposed by excess Do. do. Scarcely Insoluble 0.20 0.33 Decomposed .005 0.33 Insoluble	42.6 85.2 10.74 at 50° Sprts. sp. gr915 43.66 at 50° Sprts. sp. gr818 (Graham)
NICKET		
Acetate of Arseniate of Carbonate of Chloride of Nitrate of Protox and Ammonia Oxalate of Sulphate of Sulphate of and Ammonia and Potassa . and Iron . Tartrate of	Very soluble Soluble (Ure) Insoluble Soluble in hot water 50 Soluble Insoluble Nearly insoluble 33.3 185.71 25 11.1 Soluble Very soluble	Soluble

Name of Salt.	Solubility in 100 parts Water	Solubility in 100 parts Alcohol
	at 60° at Boiling point.	at 60° at Boiling point.
PLATINUM.		
Protochloride of . }	Soluble }	Easily soluble, also in Ether
Protochloride of .	Soluble	Insoluble
and Potassium .	Soluble	Insoluble ·
and Sodium .	Uncrystallizable	Very soluble
and Ammonium	Very sparingly	and the second
and Potassium .	Very sparingly	
and Sodium .	Soluble	Soluble
Protonitrate of	Soluble	1 1 mm
Pernitrate of	Soluble	
Protosulphate of	Soluble	(Vour coluble also to
Persulphate of	Very soluble	Ether
POTASSA.		
Acetate of	100	200
Ammonio-oxalate of .	Soluble	
Ammonio-sulphate of .	13	
Ammonio-tartrate of	very soluble	
Antimonite of	Soluble	
Arseniate of	Uncrystallizable	3.75
Binarseniate of	18.86 at 40°	Insoluble
Arsenite of	Uncrystallizable	
Benzoate of	Very soluble	
Bibenzoate of	Soluble	
Camphorate of	1 25	-
Carbonate of	100	
Bicarbonate of	25 83	
Chlorate of	6.03 60 at 1881°	- · · · ·
Chromate	48 extremely	Insoluble
Citrate of	Very soluble	
Columbate of	Uncrystallizable	
Ferrocyanide of	33.3 100	
Iodide of Potassium .	143 at 65° (G. Lussac)	Sparingly
Iodate of	Soluble	
Mulybuate of	Solubic	(0.000
Chloride of Potassium .	{29.21 at 66.83° 59.26 at 229.28° }	$\begin{cases} 2083 \\ 4.62 \text{ at } 80^{\circ} \\ 1.66 \\ . \\ . \\ . \\ . \\ . \\ . \\ . \\ . \\ . $
	( 29.31 at 64°)	(0.00 (202).834
Nitrate of	236.45 at 207° 5	2.083
	(285. at 238°)	2.000
Oxalate of	$\{50 (Ure) \}$	\$ 2.76 at 80° Sp. gr900
Binoxalate of	(10 Brande) (Ure 100)	(1 Of Spris. 8/2

Name of Salt.	Solubility in 100 parts Water	Solub	ility in 100 parts Alcohol
	at 60° at Boiling point.	at 600	at Boiling point.
POTASSA.			•
Quadroxalate of Phosphate of Diphosphate of Biphosphate of Hypophosphite of	66.66 Difficultly soluble Soluble in hot water Very soluble Very deliquescent	Very	2.91
Hyposulphate of Hyposulphite of and Silver Succinate of Sulphate of	{ Difficultly soluble at 60° readily at 212° Deliquescent Difficultly Very soluble {10.57 at 54° 26.33 at 214° 50 at 40°		
Bisulphate of Sulphite of Tartrate of Bitartrate of Tartrovinate of Tungstate of Nitro-tungstate of	200 at 220° 100 100		0.416 2.91
SILVER.	1.070		
Acetate of Arseniate of Arseniate of Borate of Chlorate of Chlorate of Chlorate of	Very difficultly soluble Insoluble Difficultly soluble 25 (Chenevix) Very slightly Insoluble Insoluble 100 200 Insoluble Soluble 1.15 Very little soluble Soluble Difficultly soluble Soluble	-	25
and Potassa .	Soluble		
SODA. Acetate of Arseniate of Binarseniate of Benzoate of Biborate of Carbonate of	35   150     {10 (Thompson)   25 (Ure)     \$25 (Ure)   50     \$30uble   50     \$033 .   50     50   100		•

Name of Salt.	Solubility in 100 parts Water	Solubility in 100 parts Alcohol
	at 60° at Boiling point.	at 60° at Boiling point.
SODA.		
Bicarbonate of Chlorate of Chromate of Citrate of Iodide of Sodium Iodate of	7.6 33.3 Very soluble 100 or more ( <i>Brande</i> ) 173 7.3	Sol. in sp. rect. Sparingly Insoluble
Molybdate of Muriate of (or Chloride of Sodium) }	Soluble Equally soluble at all temperatures $(Berz.)$ $\begin{cases} 33.3 \text{ at } 60^{\circ} \\ 100 \text{ at } 123^{\circ} \\ 50 \text{ at } 60^{\circ} & Berzel. \end{cases}$	5.8 at 80°   Sp. gr.   900     3.6   .   of   872     0.5   .   Spts.   834     (
Nitrate of	$\left\{\begin{array}{ccc} 73 & \text{at} & 32^{\circ} \\ 173 & \text{at} & 212^{\circ} \\ 80 & \text{at} & 32^{\circ} \\ 22.7 & \text{at} & 50^{\circ} \\ 55 & \text{at} & 61^{\circ} \\ 218.5 & \text{at} & 246^{\circ} \end{array}\right\} Marx$	10.5 at 80° (Sp. gr.).990 6 of .872 0.38 (Spts.).334
Oxalate of	Sparingly soluble 25 50 Soluble	
Biphosphate of Hypophosphite of Succinate of Sulphate of (Cryst.)	Very soluble Very soluble Soluble { 48.28 at 64° 322 12 at 91°	Very soluble
Sulphate of (dry)	{16.73 at 64° 50.65 at 91° 42.65 at 91° Lussac)	Insoluble
Hyposulphate of Bisulphate of Sulphate of and Ammonia	41.6 91- 50 Soluble	Insoluble
Hyposulphite of Tartrate of and Potassa	Deliquescent 56.37 (Thomson) . 20	Insoluble Insoluble
Tartrovinate of	Soluble	Sol. in sp. rect., but sparingly in absolute alcohol
Tungstate of	25 50	
STRONTIA. Hydrate of Acetate of Arseniate of Borate of Carbonate of	$\begin{cases} 0.625 \text{ at } 60^{\circ} \\ 5 \text{ at } 212^{\circ} \\ 2 \\ 50 \\ \text{Very soluble} \\ \text{Sparingly soluble} \\ \text{Sparingly soluble} \\ 0.76 \\ 0.0651 \text{ at } 212^{\circ} \end{cases}$	
Chlorate of Chloride of Strontium . Chromate of Citrate of	Very soluble 50 Insoluble (Brande) Soluble	Soluble Soluble

Name of Salt.	Solubility in 100 parts Water	Solubility in 100 parts Alcohol
	at 60° at Boiling point.	at 60° at Boiling point.
STRONTIA.		
Ferrocyanuret of	25	
Iodide of Strontium	Soluble	
Indate of	25	
Nitrate of	113	
Ovalate of	0.52	
Phosphate of	Insoluble	
Phoenbite of	Soluble	
Hypophosphite of	Very soluble	
Succipate of	Soluble	
Subbate of	0.096 at 9190	
Unpaulphite of	20 (Gay Lussac)	Insoluble
Hupogulabate of	99 99 66 66	Insolubic
Transformation of	0.67 at 1700	
Tartrate of	0.07 at 1700	
TIN.		
	0.1.11	
Acetate of	Soluble	
Arseniate of	Insoluble	
Borate of	Insoluble	-
Nitrate Proto. of	Uncrystallizable	
Nitrate Per. of	Scarcely	
Oxalate of	Soluble	
Phosphate of	Insoluble	
Succinate of	Soluble	
Sulphate Proto. of	Crystallizable	
Sulphate Per. of	Uncrystallizable	
Tartrate of	Soluble	
and Potassa .	Very soluble	
ZINC.		
Acetate of	Very soluble	
Antimoniate of	Very sparingly	
Borate of /.	Insoluble	and the second se
Chromate of	Sparingly	and the second se
Citrate of	Scarcely	and the second s
Chlorate of	Very soluble	
Chloride of	Very soluble	100 at 5410
Iodide of	Soluble	
Iodate of	Difficultly soluble	
Lactate of	2 (Ure)	
Nitrate of	Deliquescent	
Molybdate of	Insoluble	
Oxalate of	Nearly insoluble	-
Phosphate of	Uncrystallizable	
Succinate of	Soluble	
Sulphate of	140 (Dumas)	
Sulphite of	81.81 at 220°	Insoluble
Hyposulphite of	Soluble	Soluble
Sulphate of and Nickel .	33.33	1
Tartrate of	Difficultly soluble	
Tartrovinate of	Soluble	Sparingly soluble
Trisulphate of	Soluble	

#### SOLUBILITY OF ACIDS, BASES, ETC.

Name of Salt.	Solubility in 100 parts Water	Solubility in 100 parts Alcohol
	at 60° at Boiling point.	at 60° at Boiling point.
ACID.		
Arsenious		
Vitreous Opaque	1.78 (Graham) 9.68 2.9 (Graham) 11.47	
Benzoic	$\begin{array}{cccc} .50 \\ 3.9 & 33.3 \\ 133.33 & 200 \\ 5 & 33.33 \\ 11.5 \end{array}$	20 at 176 <sup>5</sup> (Henry) Soluble
Succinic (Cryst.)	4 33.33 150 (Brande) 200	74 at 176 <sup>5</sup> Soluble
Brucia Cinchonia Morphia Quinia	.1177 0.2 Insoluble 0.04 Nearly insoluble 1 Nearly insoluble 0.5	Soluble Partially soluble Very soluble
Strychnia	0.04 (Graham) 0.15	5. sp. gr. of spts. 870
Camphor Cane Sugar	0.229 200	75 at 176°

#### SOLUBILITY OF ACIDS, BASES, &c.

## CHAPTER XX.

### MACERATION. — INFUSION. — DECOCTION. — DIGESTION. — BOIL-ING. — DISPLACEMENT.

MACERATION.—The soaking or steeping of a substance in a liquid, at the ordinary temperature, is termed maceration. It is almost exclusively applicable to organic substances, being most frequently resorted to as a means of hastening and facilitating the after solution of the extractive parts of hard, compact or impervious wood, roots, stems and leaves by the more active methods of DISPLACEMENT or of EBULLITION. It is employed when the soluble principles are alterable by heat: and is also made use of to effect the solution of a substance containing several principles, the solubility of which varies with the temperature applied, as it leaves those which are not taken up in the cold to be acted upon by the aid of heat. Thus, for example, in the treatment of most vegetable substances, starch which is generally present and is only soluble at the boiling point of water, will remain untouched, while all other principles soluble without heat can be separated from it.

The mode of performing the process is merely to place the solvent and the substance to be dissolved, together in a vessel, and to allow them to remain a longer or shorter time, according to the nature of the substance. For ordinary purposes a loosely covered pan of blue stone-ware is very convenient. In delicate operations a beaker glass, Fig. 254, or solution jar, Fig. 252, is more appropriate. When the solvent is volatile, a wide mouthed stoppered bottle may be used.

INFUSION.—This process is likewise applicable almost solely to organic substances. Instead, however, of the solid remaining in contact for a length of time with the solvent, the latter is first heated to boiling and then poured upon the former. After having cooled, the liquid may be decanted or pressed out—p. 320, Fig. 276.

This mode is used for the exhaustion of flowers, leaves, roots, seeds and other substances of delicate texture, which are easily penetrable and readily yield their soluble matters; and especially for the purpose of extracting volatile ingredients. The heat applied to the solvent increases its energy; but as the material is only in contact for a limited time, the interval between the commencement and completion of the operation is not sufficient to affect the material or solution, even though one or more of its components are alterable by heat.

In pharmaceutical operations, this process is generally conducted in cast iron flask-shaped vessels with handles, enamelled on the inside and fitted with a tight cover, which is to be kept in its place from the addition to the cooling of the solvent. For small operations, a beaker glass covered with a capsule, or a yellow earthenware stew pan with lip and cover, such as can be had at the crockery shops, are admirably adapted.

DECOCTION.—This mode of solution, which is so important to the Pharmaceutist, is chiefly employed for the purpose of exhausting those vegetable substances, the components of

#### SOLUTION BY DIGESTION.

which will not readily yield to other means. It is merely an extension of the last process, and consists in that contact of the material to be dissolved with a hot solvent in a covered vessel, which is continued until all soluble matter is taken up. Most volatile matters are expelled by decoction; but those which are insoluble, save by prolonged action of heat, are dissolved or suspended, as it were, by favor of other principles present.

Decoction is only used with liquid solvents which are not decomposable by heat.

In all of the preceding processes, as well also in others in



which solid vegetable matter is subjected to the solvent action of liquids, the cullendered ladle, Fig. 253, of tinned wire is most useful for transferring the residue to the press, Fig. 276, for removal of any retained liquid.

DIGESTION.—This mode of solution differs from maceration in requiring the assistance of heat, and consists in exposing a body to the prolonged action of a liquid in a covered vessel, at any temperature between 90° F. and several degrees less than the boiling point of the solvent. The method of heating varies with circumstances, and can be by a gentle fire, or by the sand, steam, water or saline *Bath*, as the nature of the operation requires.

In analysis, glass or platinum vessels are used; but in less important operations those of other materials are more convenient and economical.

A very important advantage of digestion is, that it allows the perfect solution of all soluble portions of a substance, without modifying the nature of the solvent. It is especially useful for the decomposition of ores, minerals and other substances difficultly acted upon by acids or other solvents, and also for effecting the synthesis of compounds requiring a longcontinued heat. Moreover, it is very available in preparing alcoholic and aqueous solutions, medicinal oils and other pharmaceutical products. In analytic operations, digestion is performed in beaker

Fig. 254.

glasses. These are bell-shaped vessels, Fig. 254, of Bohemian glass, and uniformly thin throughout, so as to support a considerable elevation of temperature. The glass must be well annealed, hard and free from lead, so as to resist the action of acids. These vessels come in nests of different numbers. Their size varies gradually upwards from an ounce in capacity to a gallon.

The substance to be acted upon, in a state of fine powder, is transferred to the

glass, which must be perfectly clean, and is then mixed with the proper quantity of acid or other liquid by shaking the glass after the addition, or by the use of a glass stirrer, taking care, however, in this last instance, if for analysis, to wash off adhering particles previous to its withdrawal, with a little fresh solvent. The glass is then to be covered with a square of window glass (free from lead), a porcelain capsule or watch glass, whichever is most convenient, so that the volatilized vapors condensing upon their bottoms may fall back again into the vessel.

If the glass is small, it may be directly heated over the lowered flame of a gas or spirit lamp, Figs. 27, 119, cautiously



and gradually heightened as the glass becomes heated. To modify the action of the flame and diminish the danger of fracture of the glass, a fine wire gauze b, for the diffusion of the heat, may be interposed between its bottom and the flame. Fig. 255 represents a digestion in a beaker-glass a, over a gas lamp c. For larger vessels a SAND BATH must be used.

Thin flat bottomed flasks with narrow necks and smooth tops, Fig. 256, made of hard glass, free from lead, are sold specially for this purpose; but the common sweet oil or Florence flasks are much more economical and equally convenient for operations adapted to their capacity. When it is important that not even a drop of substance shall be lost, as in analytic operations, the digesting flask should have the form shown in Fig. 257. The body is



pear-shaped, with flat bottom, and gradually tapers towards the mouth, which is lipped to facilitate the pouring of the contents.

Digestion on a small scale with inflammable liquids, must always be effected by the sand bath, so as to avoid danger of explosion from ignition of vaporized particles. The Sand Bath may then be heated over the lamp, as at Fig. 119; and in large operations by the small charcoal furnace, as at Fig. 87.

A digestion apparatus, of Berlin porcelain, adapted for a water bath, is shown in Fig. 258. Its dimensions are 7 inches in height, and 4

inches in diameter, the capacity being about 40 ounces. The projection b, is a flange for its support in the bath; a, the socket for a wooden handle, and c, a section of the cover. These vessels, made also of other sizes, are very convenient in pharmaceutical operations, for the digestion of organic matters, especially those of vegetable origin.

Fig. 258.



Digestion under Pressure.—The solvent power of water may be greatly increased by presenting it to the substance in the state of vapor. This property affords the advantage of making aqueous solutions of highly obstinate substances. The appropriate apparatus is termed a digester. That which Papin used for exhausting bones of their gelatin, consisted of a strong hemispherical plate iron or copper pan of small size,

with a self-keyed lid smoothly ground at the edges, which becomes steam-tight by turning it around under clamps or ears at the side. Being thus tightly adjusted, after having received its charge, all steam is confined, save that which escapes by the safety valve placed at the top for the prevention of explosion. The lever attached to the valve allows the regulation of pressure according to the amount of weight applied.

The efficacy of digesters is owing to the boiling point of fluids being increased by pressure. When the above vessel is heated, the steam generated and filling its upper and vacant portion, exerts a pressure upon the surface of the liquid beneath, and by thus retarding further ebullition, causes, to a certain extent, an accumulation of heat therein.

In large operations, D'Arcet's apparatus (see *Encyclopedia* of *Chemistry*, article GELATIN) is much used. It is shown in Figs. 259 and 260. Our description refers to the extraction of gelatin from bones by water in a state of tense vaporization.



Fig. 259 is a vertical section of the apparatus. A is an hermetically closed cast-iron cylinder, into which the steam

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is conducted; a the main steam-pipe; b a vertical pipe conveying the steam into the cylinder A; c c branch-pipes leading the steam to the bottom of the cylinder; d a stopcock upon the pipe b, for regulating the entrance of the steam into the interior of the cylinder. (The tubes and the cylinder should be wrapped around with woolens, so as to retain their heat and prevent their cooling.) e is the stopcock for the discharge of the gelatinous solution; f the cover of the cylinder, which is fastened to the cylinder, so as to prevent the escape of any of its contents; g a tubulure in the cover for the reception of a thermometer; h a tub to receive the solution as it is formed; i a gutter for conveying into another vessel the grease which is run off in the commencement of the operation; K another gutter, moving on a pivot, which receives the liquid as it runs from the cock e, and empties it into the tub h, or into the trench i; l a tube for feeding the interior of the cylinder with fresh water; m a movable adjustment attached to the pipe lfor regulating the quantity of water and preventing a too great elevation of temperature in the interior of the apparatus.

Fig. 260, elevation of the interior basket, made of wirecloth. This basket, or cage, receives the cleansed and crushed bones, and is enclosed in the cylinder A; a is the handle with which, by means of a pulley, it is lifted or lowered, to be emptied or charged. Four or more of these machines make a series, and the boiler which feeds them with steam should carry a pressure of 4 lbs. to the inch. (*Encyc. of Chem.*)

When volatile or costly liquids are used as solvents, it is necessary both on the score of economy and of the efficacy of the process to use certain precautions. In making pharmaceutical preparations, of which alcohol or ether is the menstruum, they have an important bearing. For example, either of these liquids volatilizes by a high heat, and unless the vaporized particles are by a suitable arrangement condensed and returned to renew action upon the substance, the latter will be evaporated to dryness long before sufficient time has elapsed for the completion of its solution. For this purpose an ordinary cooling worm may be attached, as shown in Fig. The vapors escaping from the digesting vessel a, are 261. condensed partly in the inclined tube b, and partly in the worm c, and fall back again into the flask as soon as they become liquefied by the water surrounding the worm. This

## SOLUTION .- MOHR'S DIGESTER.

arrangement allows a prolonged contact of solids with volatile liquids, without loss or alteration of the latter-a very important consideration, as time is an influential adjunct in digestion.

Fig. 261.



Mohr improves upon the above apparatus, by substituting another, exhibited in Fig. 262. It consists of a tin plate cylinder A, tubulated at its bottom. Through this tubulure

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passes a glass tube t t, adjusted by perforated corks to the tubulures of both cylinder and digesting vessel M. The vaporized matter, ascending from the heating vessel M, is cooled by the water in the cylinder, and which surrounds the tube t t. The long barreled, tin plate funnel T, receives the amount of water freshly added, and conveys it to the bottom to displace that which has become heated, and which by its less density rises to make its escape through the outlet A.

SOLUTION BY BOILING.—This mode is resorted to when a substance can only be exhausted of its soluble portion at the boiling point of the solvent. The exact point of temperature at which a liquid boils, depends partly upon the amount and fluctuations of pressure, and the nature and construction of the vessel. When the pressure of supernatant vapor is removed by uncovering the vessel, ebullition is facilitated and takes place at lower temperatures. Indentation or roughening of the surface of the heating vessel, or any other means by which the heating surface is increased and escape of gaseous matter is promoted, lowers the boiling point of a liquid. For this latter reason, platinum scraps or pieces of unglazed card, or of cork, pacify turbulent ebullition and render the process tranquil and uniform.

The heat applied should never exceed the degree at which the solvent boils, especially in metallic vessels, otherwise ebullition is retarded, for beyond a certain temperature a repulsion between the particles of liquid—when water is used—and the metallic surfaces, prevents contact.

The kind of apparatus varies with the nature and quantity of material under process.

Boiling in Tubes.—Test tubes, Fig. 263, are very convenient implements for delicate solutions, assays and the like, and, therefore, the laboratory should be supplied with a large assortment, varying from three inches in length and a quarter inch in diameter to six inches in length and one inch in diameter. They should be of hard, white German glass, free from lead, with perfectly round bottoms, uniformly thin, so as to withstand heat. The racks, Figs. 25 and 147, serve as their supports.

A test tube should never be charged with more than onethird its capacity of solvent, else there may be loss by ejection from too sudden ebullition; and the solid substance previously powdered is not to be added until the liquid is brought to boiling, and then only in small portions at a time.

To guard against spirting or to ensure a uniform heating, the tube must be gradually heated, not upon its bottom but near to or on the side, as shown in Fig. 264. It is, as seen, heated over the small lamp, Fig. 115, being held in the fingers, which are protected from contact with the hot glass



by a doubled strip of thick paper wrapped around the neck of the tube for its support. The spring holder, Fig. 265,



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consisting of a wooden handle affixed to two flat pieces of thin steel indented at their ends so as to form a round catch, and tightened or loosened by a slide, is much more convenient but not always at hand.

The mouth of the tube during heating, or whilst its contents are being shaken, should always be held away from the operator, else ejected matter may endanger his person or dress.

Faraday gives the following valuable advice as to the use of test tubes for making solutions with volatile liquids, and under pressure.

"In consequence of the small diameter, and therefore small sectional area of tubes, they are much stronger relatively to internal pressure than larger vessels, such as flasks of the same thickness. An advantage is thus gained in some cases of solution or digestion in certain fluids, as alcohol, ether, and even water, because it enables the experimenter to subject the substances to temperatures as high as the boiling points without loss of the fluid, or occasionally to temperatures still higher, the ebullition going on as it were under pressure. This is easily performed with alcohol, ether, and similarly volatile fluids, in tubes of four, five, or six inches in length, and of such diameter as to be readily and perfectly closed by the finger. Suppose a tube of this kind, onethird filled with alcohol and held tightly between the thumb and second finger of the right hand, its orifice being closed by the fore-finger of the same hand. Fig. 251. The fore-finger is to be relaxed, and the heat of a spirit-lamp applied until the alcohol begins to boil; the fore-finger is then to be reapplied closely, and it will be found that the flame of the lamp, applied at intervals, is quite sufficient to keep the temperature up to the boiling point. No alcohol can evaporate, for the finger has power sufficient to retain the vapor even were its force equal to two atmospheres, and the tube itself is also strong enough to resist the same force.

"This operation is very advantageous when valuable and volatile solvents are in use; it is therefore worth while to refer to those points which indicate the state and temperature of the fluid, and which make the practice easy. If the fluid be one which, like alcohol, when at or above its boiling point is at a temperature inconvenient to the hand, then, if all the common air were allowed to pass out of the tube before closing it, the whole tube would become heated by the vapor rising from the hot liquid beneath, and the fingers would be injured; but by not allowing all the air to escape, that portion which is retained in the tube, is always forced to the top by the successive formation and condensation of the vapor below, and interfering with the passage of the hot vapor to the part which it occupies, it preserves that portion of the tube at comparatively low and very bearable temperatures. The part thus retained at a low temperature, is proportionate to the quantity of air confined in the tube; this quantity is usually a proper one if the tube be closed just after the alcohol has begun to boil, and before the upper part of the tube has been heated. If too much air has been expelled, and the tube is found to become hot above, the application of the flame must be suspended a moment or two, the whole suffered to cool below the boiling point, the tube opened, the upper part cooled slightly by a piece of moist paper or a cold finger, and then the fore-finger is to be reapplied to close it as before.

"The state of the fluid within is in part indicated by the pressure of the air or vapor on the finger, the latter being urged away from the tube by a force proportionate to the degree of heat above the boiling point, and being drawn inwards when the heat is below that point. Generally, therefore, the finger alone will serve to ascertain whether the temperature is above or below the point of ebullition; but as the force required is, after operating for some time at high pressures, such as to diminish the sensibility of the finger to smaller pressures, it sometimes happens that on lowering the temperature, the period at which it attains that of ebullition in the atmosphere cannot be distinguished. This point is, however, easily recognized by relieving the pressure of the finger slightly; should the quiescent fluid below then burst into ebullition, it is a proof that its temperature is higher than the boiling point at atmospheric pressure, but should it remain quiescent until the finger is entirely removed, its temperature will be known to be below that point."

Boiling in Beaker Glasses and Flasks.—These vessels are used when large quantities of liquid are to be operated upon. When the direct heat of the lamp is applied, it should be diffused by the intervention of a wire gauze. The preferable mode of heating is by a highly heated sand-bath. The same remarks as to their material and construction, as given before

### BOILING IN CAPSULES.

at p. 303, are applicable in this instance. They should be loosely covered during the operation, the beaker glasses with squares of window glass and the mouths of the flasks with watch glasses. The position of the beaker glass over the lamp is shown at Fig. 255, that of flasks at Fig. 119. Round bottomed flasks, Figs. 266, 267, are made of different sizes, espe-



cially for solutions, but Florence flasks, which have been rounded on the edges of the mouth over the blow-pipe flame, so as to allow of the easy entrance of a loose cork, are equally convenient and less costly. They are thin and uniform throughout, and bear very high temperatures without fracture. Fig. 268 represents a flask being heated in a dish sand-bath over a small furnace, for solutions requiring a higher temperature than can be furnished by the gas or spirit lamp. They must be well imbedded in the sand in order to produce ebullition.

Boiling in Capsules.—Solution is made in open vessels when the solvent liquid is not easily vaporizable or alterable by exposure, or when its loss is of little consequence. The most convenient implements for the purpose are porcelain capsules. Figs. 269, 270. Those from the Royal, Dresden



Fig. 270.

and Berlin factories are far superior to the French, or those of any other make. They are strong yet uniformly thin throughout, and support very high temperatures and sudden changes. Being enamelled they are protected from the action of acids or corrosive liquids, and consequently are of general application. They are sold of all sizes, by Kent, varying upwards from an ounce to 18 oz. in capacity. The diameter of the smallest is about 2 inches, and that of the largest  $15\frac{1}{2}$ inches. The depth should be one-third of the diameter. The smaller sizes come in nests of a half dozen or more. Fig. 269 represents one with spreading rim and lip to facilitate pouring. That shown in Fig. 270 has a more hemispherical shape.

Capsules are almost always heated over the open fire, the spirit or gas lamp furnishing the requisite temperature. Those of smaller size are shown in position upon suitable supports at Fig. 118, and i, Fig. 119; for the larger Luhmé's lamp, Fig. 122 answers an admirable purpose.

The liquid is placed in the capsule before the ignition of the wick, and when it is boiling, the substance to be acted on should be gradually deposited in it in a finely divided state, while constant stirring with a glass rod is kept up. After all has been added, both ebullition and stirring must be continued until the completion of the process, taking care to supply the loss of the volatilized portion by fresh additions of the solvents, unless the solution is to be evaporated.

When the nature of the materials requires the intervention of a medium other than sand to modify the heat, a rare occurrence when operating in capsules, the latter are heated over baths, as shown at Fig. 150. Capsules or boiling pans of enamelled iron ware or tinned copper are used only in very large operations.

SOLUTION BY STEAM.—When a substance is to be dissolved by steam heat, and the nature of the materials renders the direct application of steam inadmissible, then baths, Fig. 11, come very appropriately into play.

For aqueous solutions which are greatly facilitated by the immediate action of steam, it is supplied through flexible lead tubes leading from the generator, Fig. 10, directly into the containing vessel.

For small operations in glass vessels, the copper spritz,

Fig. 186, half filled with water and heated over the gas lamp, readily furnishes sufficient steam.

As boiling by steam is practiced in numerous chemical operations, it is proper to introduce some directions pertinent to the subject.

It is very seldom that the heat required for ordinary laboratory purposes exceeds that given by five pounds above atmospheric pressure—never more than fifteen pounds—and the fire under the generator and weights upon the safety valve should be regulated accordingly.

If the mixture to be boiled is unalterable by the action of condensed steam, the conduit pipe may lead directly into it, and to the bottom.

As the liquid appears to boil before it actually does, the only sure indication of temperature is to be obtained by a thermometer, Fig. 84.

This method, however, causes a great loss of heat and incommodes the operator, by filling the apartment with clouds of vapor. A *loose* cover will partially remove these objections. In boiling in this way, care must be taken that the fire does not get low, lest a condensation of the vapor occupying the upper part of the boiler causes a partial vacuum, and the consequent withdrawal of part of the liquid from the vat into the boiler. The conduit connected with the feeder should always have a stop-cock near the coupling, which is to be shut off upon the completion of the operation. If the boiler should then happen to be surcharged with steam, it must be blown off through the valve, this being readily accomplished by gradually unloading the lever.

A far better plan of boiling by steam is to conduct it through a coil of pipe placed at the bottom of the vat, and having an exhausting pipe leading into the neighboring flue. This mode allows a uniform temperature at any degree from that of the atmosphere to 212° F.,—suitable stop-cocks being attached for that purpose to regulate the supply of steam accordingly.

In cold weather and especially when the feeder or conduit are of any length, it will be economical to wrap them with woolen listings or straw, as means of preventing condensation.

SOLUTION BY DISPLACEMENT.—Displacement, termed also lixiviation, when applied to the solution of saline substances, 21

### SOLUTION BY DISPLACEMENT.

is an economical process for the extraction of the soluble portions of woods, leaves, flowers, barks, precipitates, and, indeed, of all matters to which suitable apparatus can be adapted for the infiltration of a sufficiency of liquid through them. For delicate operations and those conducted upon a scale of moderate extent, glass vessels may be employed. One of the usual form is shown in Fig. 271. It is made of hard glass, free from lead, the upper part, or A, being the displacer, and the lower part, B, an ordinary flask, the recipient of the

Fig. 271.





The mouth of the bottle and that portion saturated solution. of the displacer which rests in it should be ground so as to make a close joint. The stopple is for closing the upper vessel when necessary. Dobereiner's improvement upon the above, but operating upon the same principle, is shown in Fig. 272. To prevent the passage of the material through the barrel of the displacer, it must be loosely closed with a plug of raw cotton as at f, and then adjusted by means of a perforated cork q, with the vertical tubulure of the globular receiver a. The whole apparatus as adjusted is retained in an upright position by a support, the receiver resting upon a braided straw ring. It is now ready to receive its charge. substance in coarse powder and moistened, occupies the part of the vessel e, and the solvent is subsequently added as at d. A partial vacuum being made in the receiver by the evaporation of a few drops of alcohol added through the lateral stoppered tubulure c, the liquid percolates through the solid mass by the force of atmospheric pressure, and ultimately reaches the receiver saturated with the soluble matter of the material e.

In order to express clearly the rationale of this process, we will suppose that vegetable matter, a dye-wood for example, in coarse powder, is to undergo exhaustion by this method. It occupies the part e, as before said, and to facilitate the percolation, has been previously moistened with a portion of the solvent. Liquid is added, as shown at d, and soon soaks into the mass beneath; another portion of solvent is then poured in as before, and takes the same course, displacing the portion before used without mixing with it. These strata of solvent are pressed downwards by successive additions of liquid, and become more and more charged with soluble matter, as they approach the bottom of the mass, until at last they run out into the recipient highly charged and in the present instance, highly colored-the first runnings being more nearly saturated than those which follow. When by consecutive additions of solvent the material has been exhausted, the liquid in its transit through the mass is unacted upon, and reaches the receiver as tasteless and uncolored as when first poured in. This indicates the completion of the process.

The neck of a retort with its smaller end adjusted to a wide mouth bottle by means of a perforated cork, makes an excellent displacing apparatus.

When the solvent is volatile, in order to prevent loss by evaporation, the apparatus is modified, as shown in Fig. 273.

The displacer is adapted to the centre tubulure of a two necked Wolffe bottle. Now as atmospheric pressure is an important element of this process, it will not do to shut it off by closing the top of the displacer without making some other arrangement, and, therefore, a communication between the upper and lower vessel is established by means of a bent tube adjusted in the lateral tubulure of each. In this manner the vessel is completely closed, and vaporization prevented while the pressure produced is distributed throughout the vessel, and thus



rendered uniform. In using the glass displacement appa-

ratus first described, this principle must be recollected; where a vacuum is not artificially created in the receiver, the ground glass edges of it and the displacer should either be permanently separated or occasionally disjointed.

The stop-cock near the bottom of the receiver allows the withdrawal of the solution as fast as it accumulates, without the necessity of disarranging the apparatus. In experiments upon large quantities of material, and in pharmaceutical operations generally, the displacers employed are made of tinned copper or tin plate. Those of porcelain which are now in the market, are much more serviceable, because they are readily cleansed and resist the action of corrosive liquids. Of whatever material they are made, they should be cylindrical and funnel-shaped at the base, and the height should be at least four times the diameter, as is shown in Fig. 274. At a there is a flange in the interior as a support for the cullendered diaphragm a b. These diaphragms are removable, and for convenience in handling have a knob in the centre. The lower diaphragm is always to be covered with a circle of coarse muslin for the reception of the material and to prevent the passage of particles as well as obstruction of the holes. The other diaphragm, fitting loosely to the cylinder, rests upon the top of the powder and serves for the better distribution of the solvent and for the prevention of the escape of dusty particles which sometimes occurs if the powder is put in dry. The stop-cock c in the barrel or exit pipe is useful for regulating the discharge of the liquid.

The tripod D is the support, and allows the withdrawal of the receiver P, when it is full and when it is to be replaced by another, without the necessity of disturbing the displacer.

Another very convenient form of displacer is that in which ether, alcohol and any other volatile solvent may be kept in constant action without exposure to air or loss by evaporation. By its use a great saving of time and labor and solvent is gained. The arrangement is exhibited at Fig. 275. It consists of a glass cylinder B, the funnel of which should reach to the centre of a glass balloon A, beneath the two vessels, being attached by means of a perforated cork. The lateral tube c, d opens communication between the lower and upper apartments. As the whole forms a perfectly tight connection, the safety tube E becomes necessary for the regulation of the dilatation and contraction of the vapors.

# SOLUTION BY DISPLACEMENT.

When it is desired to exhaust a vegetable matter with alcohol or ether, and at one and the same operation to con-

Fig. 274.





d

required by the small spirit lamp I. The ether or alcohol which has infiltrated into the balloon thus carried to, and maintained at ebullition, passes off as vapor into the lateral tube, there partially condenses and falls upon the material in the cylinder to infiltrate through again. The excess of vapor and of expanded air escapes through the safety tube; but a part of the vapor is arrested and condenses in the three bulbs, the first of which immediately empties its liquefied contents into the cylinder to renew its action upon the powder.

The concentration of the filtered solution is thus continually going on in the balloon, the concurrent distillation returning the evaporated particles to the substances to be exhausted, through the lateral tube c, d.

When water is the liquid to be employed, the water bath must be replaced by a saline or sand bath, and the spirit lamp by a furnace.

For the solution of difficultly soluble substances, this mode presents many advantages not possessed by other processes, and amongst others it yields a clear solution and supersedes the necessity of FILTRATION, which is required for most solutions made by infusion, decoction and boiling. It is particularly applicable to the purpose of procuring concentrated solutions for EVAPORATION to extracts, as well as for making tinctures, &c.

The solvent may be acid, alkaline, spirituous, ethereal or aqueous in its nature, the principle of its action being the same in all cases. When the liquid is corrosive, however, the vessel should not be metallic, but of glass or porcelain, and should be plugged with asbestos instead of cotton. It is immaterial whether the solvent be applied cold or warm, save when the process is resorted to for the separation of substances soluble in cold from those which are only soluble in hot liquids. Except in such cases heat may be applied, as it increases the power of the solvent, and a convenient means of doing so is to encompass the apparatus with a metallic jacket, to be supplied with steam from the generator, Fig. 10.

There are certain conditions necessary to the success of this operation. The material must be in powder of medium DIVISION; neither too fine nor too coarse, for in the first case it clogs the cloth and holes of the diaphragm, and if heavy and compact, retards the percolation of the liquids: on the other hand, when too gross, the transit of the solvent is so rapid that the material is but partially acted upon. When alcohol or ether is used, the powder may be a little finer than for less volatile solvents; and all powders liable to *set*, or to become so compact as to prevent the passage of liquid, must previously be mixed with well washed coarse white sand. This addition remedies the defect and ensures the easy passage of the solvent.

The material, as before recommended, should be moistened with half its weight of the solvent, and left to soak for an hour or more before being placed in the percolator. After having been transferred, it is covered with the diaphragm, and sufficient liquid is poured upon it to cover entirely its surface. As soon as this first portion infiltrates through the mass, another portion is added, for it is only by keeping the surface covered with solvent that a uniform penetration of all portions of the mass can be effected. If the liquid passes through very rapidly the mass is too loose, and must therefore be compressed by pressing upon the diaphragm cover. Or in order to prolong their contact, the stop-cock c may be nearly closed, so as to allow the exit of only a thin stream.

When alcohol or other valuable volatile liquid is used, the residual portions may be either extracted by pressure of the mass P, or by displacement with water; and subsequently, by distillation of the resulting mixture. The general practice, however, in the Laboratory is to reserve the last running for the first application to fresh material.

CADET'S MODE OF SOLUTION—This plan is well adapted to those powders which do not admit of being easily infiltrated. It consists in macerating or infusing the pulverized material with double its weight of cold or hot solvent, and after some time subjecting it to strong pressure. This treatment is to be repeated until the substance ceases to yield soluble matter, and the resulting liquids are then mixed together and filtered. Cadet's mode is used largely for dissolving the tannin from galls with ether.

A convenient press for this purpose is shown at Figs. 276 and 277. All powders which have undergone the process of solution in large quantity should be subjected to its action, as a good deal of retained solution may thus be obtained, and consequently saved.

It is formed of two strong upright stanchions, and two proportionably strong cross pieces, firmly jointed in the side beams. The upper cross piece carries a box through which works an ordinary press screw, in the usual manner. Upon the lower cross piece, is placed a wooden trough, A (Fig. 277)



at least two inches deep, and to the front of which is adapted a gutter B for the conveyance of the liquid, which assembles in the trough, to a vessel E placed at and beneath its mouth. Upon and within the trough is placed a wrought iron plate This cylinder is formed of two semi-cylinders cylinder. joined together. Throughout its height, it is divided off alternately into equal parts by zones or belts. The zones a a are more than an inch broad; -- the partition, b, &c., four or five inches in width. The top as well as the lower zone is narrow. All the wider divisions are cullendered throughout their circumference with innumerable small holes, through which the liquid is to flow when pressure is applied. All the narrow zones are secured by a strong wrought iron ring, formed of two pieces working on a hinge adjusted at the back. Upon the front is a movable broach D, which bolts them together, and makes the cylinder compact, so that it can resist the pressure applied. When the marc is exhausted, by draining out the broach D, the circumference of the cylinder is loosened or extended, so that its contents can be removed without The marc is placed in this cylinder and pressed difficulty. out by the power of the screw, until no more fluid will exude, even with the force of a man to the lever. The liquid runs into the gutter B and through a sieve, which should be properly placed for the purpose, into the vessel E, and may thence be drawn off after it has settled, into suitable vessels. The

## SOLUTION UNDER PRESSURE OF STEAM.

residual exhausted powder is, as said above, easily emptied out by loosening and removing the pin D.

Solution under Pressure of Steam.—Figs. 278 and 279 exhibit Duvoir's bucking apparatus, which as modified in the



drawings, is applicable to the exhaustion of organic matter. B B are the wooden vats lined with lead which receive the material to be displaced; G G the cullendered diaphragms for its support; and c c their movable covers counterpoised so as to admit of ready depression or elevation at will. These false bottoms are also movable, so as to afford facility in cleansing the vat, and they should, when in use, be covered with crash cloths to prevent obstruction of the holes. The tubes D D, communicating with the steam generator, Fig. 10, traverse the centre of the vats, and are surmounted by metallic discs, E E, for the reverberation of the vapor rushing against them.

The directions for the management of the apparatus are nearly the same as for displacement generally. When the vat has received its charge, the cover is to be lowered and fastened down by clamps, and steam let on by opening the stop-cock of the feeder. As the steam generates, it passes over through the pipe D, reaches the disc E, and is projected uniformly over the whole surface of the material. The elastic force of the vapor accumulating in the upper portion of the vat exerts a pressure upon that portion which has condensed and forces it downwards through the mass. In its passage it becomes

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charged with soluble matter and reaches the lower part of the vat  $\kappa$  beneath the diaphragm, whence it is drawn off through the cock L, R.

As a safeguard against accidents there should be a safety valve upon the cover of the vat as well as upon the generator.

This arrangement would be particularly economical in the arts, for extracting dyewoods and other vegetable substances.

# CHAPTER XXI.

## EVAPORATION.

WHEN any liquid is heated for the purpose of expelling vaporizable matter, and the process is conducted solely with a view to saving its fixed portion, the operation is termed evaporation. It thus far differs from distillation, which has for its object the preservation of the volatilized portion, in most cases, regardless of the solid. By its aid we can decrease the volume of, or concentrate solutions for *crystallization* and chemical reaction, expel valueless volatile ingredients from those which are more fixed, obtain dissolved matter in a dry state, and prepare extracts and other pharmaceutical products.

Liquids evaporate more or less at all temperatures, those having the lowest boiling point yielding the most readily; but there are certain conditions which greatly promote this tendency. It must be remembered, therefore:—

1. That evaporation is more rapid in dry atmospheres, and that consequently the transit of a constant stream of air over the surface of the heated liquid effects a continual removal of each stratum as it becomes saturated with vapor.

2. That evaporation is confined to the surface, and consequently that the breadth of the evaporating vessel must be extended at the expense of its depth.

3. That heat greatly facilitates evaporation by lessening the cohesive force of the particles of a liquid, and consequently that the evaporating vessel should present a broad surface to be heated.

## EVAPORATING VESSELS.—SPONTANEOUS EVAPORATION. 323

4. That a diminution of the atmospheric pressure also facilitates evaporation, for the more perfect the vacuum the lower the boiling point of a liquid.

Evaporating Vessels.—For analytic purposes, capsules of Berlin porcelain are by far the best implements. The capsules should be very thin, with steep sides, spout for pouring, nearly flat bottomed, and glazed throughout. Watch glasses answer for small experiments, but require to be very cautiously heated, as they are readily fractured.

Beaker glasses are also used for evaporating solutions which would lose by being transferred. Broad mouthed glass flasks are of but limited application for evaporating, and are only employed for slow processes with valuable liquids, which are liable to alteration by too much exposure when ebullition is necessary.

For the larger operations of the Chemist or Pharmaceutist, vessels (Fig. 280) of copper, tin, enamelled iron, tinned copper, and for some purposes very large porcelain capsules are more suitable.

Retorts are used when the vaporized particles are of sufficient value to be condensed, as in the process of distillation.

Spontaneous Evaporation.—Those liquids which are very volatile, or which become altered by heat, are evaporated by mere exposure to the atmosphere at its ordinary temperature. To this end they are poured into broad shallow vessels, and placed aside until the dissipation of all vaporizable matters, or until crystallization; this mode of evaporation being also employed for procuring large crystals, which are better defined than those obtained by rapid evaporation.

The more dry and hot the atmosphere the more rapid is the evaporation. In order to maintain a continued contact of the surface of the liquid with strata of fresh air, the vessel containing it should be placed in a draught, so that those portions of air which become saturated with vapor may be displaced.

When the air might act injuriously, and a vacuum is unnecessary, a substance may be evaporated in another atmosphere, for instance, of hydrogen or carbonic acid. For this purpose it is only necessary to adjust the disengagement leg



of the apparatus (Fig. 219) to the tubulure of a retort, so that its end may reach nearly to the level of the liquid in the latter. The generated hydrogen passes into the retort heated to the required temperature, and promotes the discharge of the vapors into a recipient attached to the beak of the retort, and fitted with a small tube in its other tubulure for the disengagement of uncondensed portions.

For the evaporation of solutions of sulpho-bases, of sulphosalts, and of all substances readily oxidizable by exposure, this process is better applicable than that with the air-pump, which is apt to be attacked when the eliminated vapors are corrosive.

This process is much used in CRYSTALLIZATION, for concentrating alterable solutions, and drying precipitates.

Evaporation in Vacuo.—We have already referred to the happy influence of diminished atmospheric pressure in facilitating evaporation, and shall now speak of the means by which it is accomplished, and the particular instances in which it is employed.

This mode is resorted to for hastening the evaporation of all liquids, but more especially of those which are alterable by exposure.

In small experiments we use a capped bell glass (pp. 337, 338) as the confining space. Under this bell glass is placed the broad shallow capsule, with its liquid contents, supported upon a wire tripod resting in a leaden tray containing sulphuric acid, dried chloride of calcium, fused potassa, or some other absorbent material. The bottom or bed of the bell may be a ground glass plate, and to seal the joints hermetically the rim of the bell should be greased. Connection being made by means of a suitable pipe and the stop-cocks between the bell and the syringe, communication is opened and the vessel exhausted of air. The pressure being thus removed, evaporation proceeds rapidly, and until the absorbent matter becomes saturated with vaporized particles, or the bell filled, there is The latter can be partially removed by no impediment. working the pump at frequent intervals.

When an air-pump is used the procedure is the same, but in either case the vacuum must be produced gradually, otherwise the sudden ebullition of the liquid may cause ejection of its particles. The better way is to cease pumping as soon as the barometer attached to the machine indicates from two to two and a half inches pressure, and to resume the process of exhausting again at intervals of fifteen or thirty minutes.

Other modes of evaporating in vacuo, as practiced in the arts, are fully described in Ure's Dictionary of Arts, and under Sugar, in the "Encyclopedia of Chemistry." Howard's and Barry's vacuum pans are the most effective implements. The latter is applicable in Pharmacy for making extracts upon an extensive scale. It consists of a hemispherical pan with a tightly fitting cover, in the centre of which is a bent tube leading into a copper spheroid of four times the capacity of the pan. This tube is fitted with a stop-cock, which allows a communication, at will, between the spheroid and pan. Another cock at the opposite end is made so as to couple with the conduit of the steam generator.

The liquid to be evaporated is introduced into the basin, which is then to be hermetically closed and placed in a water bath. The cock connecting with the spheroid being closed, a current of steam is let on, and continued until the entire expulsion of air from the pan; access of steam is then stopped by closing the cocks, and a sheet of cold water applied to the exterior. A condensation of vapor ensues, and a partial vacuum is produced. Communication being then opened with the caldron, uniform expansion of the air ensues; and as the capacity of the spheroid is four times greater than that of the pan, the latter contains only one-fifth of its original amount of air. Several repetitions of this manipulation produce a sufficient vacuum. The water bath is then heated until the liquid within the pan commences to boil, as may be seen through the small window left for the purpose, and the cooling of the spheroid continued. When the liquid has reached the required thickness, the operation may be discontinued. In this way ebullition proceeds at 100° F. under a pressure sixteen times less than that of air. With an air syringe attached for removing the vapor as fast as formed, the power of the apparatus would be greatly increased.

Evaporation by Heat in Open Air.—Having already noted the effects of heat in facilitating evaporation, we proceed to make known its modes of application. As the boiling points of solutions differ, so accordingly their evaporations are effected at varying temperatures. For example, aqueous or other solutions of unalterable matter may be evaporated over the fire; others which are destructible by heat require the intervention of BATHS. In whatever mode the operation is performed the general principles are the same, and whether the vessel be a porcelain capsule or metallic pan, the greater its width in proportion to its depth the more rapid is the evaporation. Constant agitation with a stirrer is also promotive of the process.

Evaporation over Water and Saline Baths.—When solutions are alterable at a temperature above 212° F., the capsule or containing vessel is heated over the WATER-BATH, Fig. 150.

If it requires a higher heat, but one not exceeding 300° F., then the water must be replaced by a SALINE-BATH, p. 184.

Evaporation by Steam.—This mode has many advantages over all others, not among the least of which is that with the aid of the generator, Fig. 10, any number of vessels may be heated simultaneously, and in any part of the laboratory, it being only necessary to have conduits of sufficient length to convey the steam to them. Moreover, convenient stop-cocks allow a regulation of the heat, and consequently all danger of injury to the evaporating solution is avoided. By increasing the pressure of the steam the temperature of the solution is also elevated.

Steam is applied through metallic coils placed at the bottom of the containing vessels, and having an exit pipe leading into the neighboring flue, or else by means of metallic casings. This latter mode, by far the best, is given in detail at pp. 42 and 181.

Evaporation over Sand-baths.—This mode is much used in analyses and for careful evaporations, requiring temperatures greater than 212°, and yet not so high as those given by the naked fire. The position and arrangement of the vessels are as directed under the head of SAND-BATHS.

Evaporation by Heated Air.—This mode is admirably adapted for the inspissation of the natural juices of plants or for preparing dry extracts. It is also applicable to the completion of evaporations which have been carried as far as is safe over the naked fire. Porcelain plates or panes of window glass are the vessels used, and a stove or apartment for their reception heated from 95 to 110°, with a free draught passing through are the means of obtaining the required temperature. The juice evaporates either to thin scales or else to a spongy mass, as in the case of tannin extracted by ether,

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and as soon as it reaches dryness, the plates or panes are to be withdrawn, and their contents removed with a spatula.

Evaporation over the Naked Fire.—The tendency of many substances to decomposition over fire, especially organic, even when in solution, renders this mode inapplicable save when the solvent and substance dissolved are both inalterable below the boiling point of the former. It is resorted to for expediting evaporations, but otherwise is far more inconvenient than steam, because of its affording less facility for the regulation of the heat and requiring greater attention. The containing vessel should be placed over a furnace of small dimensions, and its contents continually stirred with a porcelain spatula—this precaution preventing decomposition or carbonization, provided the temperature is not allowed to exceed the boiling point of the solvent.

In analysis and other processes, the heating implement is generally the gas or spirit lamp, Figs. 26, 27. The capsule filled to about two-thirds its depth with liquid, being placed in position, the flame is applied gradually and maintained just low enough to prevent ebullition; and in order to facilitate the process, and at the same time to allay turbulence, it should be frequently stirred with a glass rod. The same directions apply when the operation is performed in a beaker glass, as is done in some analytic experiments; and Fig. 255 shows its position over the lamp.

A cover of white paper prevents access of dust without retarding the process, but care must be taken that the contents of the vessel be not ejected against it, thus causing a loss.

In evaporating to dryness, towards the end of the process the flame must be so managed as to impart a uniform heat to all parts of the thickened solution. The interposition of a very thin plate of sheet iron between the flame of the lamp and the bottom of the heating vessel is an additional means of preventing spirting. These precautions and constant stirring will prevent the loss of particles which is liable to occur upon the disengagement of the last portions of liquid. If the liquid drops a powder during the operation, the vessel must be inclined, and in order to prevent spirting, heated above the deposit.

A platinum spatula is a very useful implement for detaching any efflorescent matter which may "travel up" the sides of the yessel.

# CHAPTER XXII.

### CRYSTALLIZATION.

WHEN a body in the act of passing from a liquid or gaseous to a solid state arranges itself in symmetrical forms, the process is termed crystallization, and the parts of the body so aggregated are called crystals.

By this process we can separate crystallizable from amorphous substances dissolved in the same menstrua; purify crystals from foreign and coloring matters, and in qualitative examinations, be enabled to determine the composition of bodies by a reference to the characteristics of figure.

The modes of crystallization are by FUSION, SUBLIMATION, SOLUTION and CHEMICAL REACTION.

Crystallization by Fusion.—Sulphur, lead, bismuth, tin, antimony, silver, numerous alloys, anhydrous salts and other fusible substances which are unalterable by heat are crystallizable by FUSION.

To this end they are melted at the lowest possible temperature, and allowed to cool very gradually. As soon as a crust forms upon the top, which may be readily seen by the surface becoming furrowed, it must be pierced with a rod and the still fluid portion decanted with sufficient dexterity to prevent it from cooling during the process, and at the same time from injuring the crystals coating the interior of the vessel.

The liquid matter should be placed so as to be free from all vibration. The greater the mass of the material and the more slowly it is cooled the more voluminous and better defined will be the crystallization.

Crystallization by Sublimation.—Volatile solids, as iodine, camphor, several metallic chlorides and mercurial compounds, arsenic, benzoic acid, iodide of lead, &c., when heated as directed in SUBLIMATION, yield vapors which, in cooling, take the form of crystals.

Crystallization from Solution .- When it is desired to ob-

tain a substance in crystals, it must first be liquefied or made into a SOLUTION with an appropriate liquid. If, after making the solution, there be any insoluble residue it must be separated by FILTRATION; and subsequently, if the solution is capable of decolorization by such means, it should be boiled with a small portion of clean bone or ivory black, and again filtered. As it is the almost universal law that heat increases the solvent power of bodies, the solution should generally be made and clarified at the boiling point, so that the excess of matter taken up at the high temperature may separate on cooling in the form of crystals.

So long as a solution is dilute it yields no crystals;—these latter are only formed when the containing liquid is supersaturated, or, in other words, holds more than it can retain; and consequently in diminishing the quantity of the liquid by EVAPORATION, we increase the density of that which remains, and hence, upon cooling, it deposits that excess of the dissolved substance which it only held by virtue of its high temperature.

Some substances are so easily soluble, and to such an unlimited extent, that their solutions form crystals immediately upon cooling; others again are taken up with such difficulty, even at high heats, unless in large bulks of liquid, that although exposed to prolonged ebullition they require to be evaporated in order to separate what has been dissolved. As the mode of evaporating has an important influence upon the form and size of crystals, we give some hints as to the proper manner of performing it.

If large and well defined crystals are required, the solution should be subjected to spontaneous evaporation, for the more slow and uniform the concentration, the more regular and gradual will be the superposition of material required to make distinct and large crystals. A slight addition of solution of gelatin will, in some instances, it is said, give the crystals the form of plates, as in the case of boracic acid.

The solution should be removed from the fire as soon as drops, withdrawn by a glass rod and deposited upon a watch glass or clean spatula, give small crystals upon cooling. If, however, a very dense crystallization is required, the concentration may be continued until a pellicle forms upon the top, but then the solidified masses are confused and less brilliant. These essays indicate that the liquid is evaporated to a point at which it cannot retain all of its soluble matter. The vessels are then placed aside to cool gradually and uniformly, that the excess may crystallize out of the liquid. The temperature should be regular, for slight variations may alter the form of the crystals.

Bodies equally soluble in cold and hot water, as well as those which are deliquescent, require a prolonged evaporation as they only crystallize from very dense solutions.

When the liquid is to be converted *wholly* into solid, then the process is termed *granulation*, and is practiced by concentrating it to a syrupy consistence, removing the vessel from the fire and stirring it *constantly* until the mass has cooled into granules. This mode is adapted for purifying pearl-ash and converting it into *sal tartar*, and also for graining brown sugars.

If the liquid, evaporated as above directed, becomes colored or murky during the process from partial decomposition, it may be treated with bone black, and again filtered into a capsule, or other vessel, previously warmed by a rinsing with hot water, so as to prevent confused crystallization from sud-The blue stone-ware capden contact with its cold surfaces. sules, which are made of suitable forms by Mr. Perrine, of Baltimore, are far better than porcelain capsules or glass beakers, as they are not only more durable, but by the roughness of their interior surfaces far more promotive of crystallization. Stone basins for this purpose, called crystallizers, are made of all sizes, in depth greater than in breadth, and with a lip to facilitate the separation of the residual liquid from the crystals. This residual liquid, called the mother water, is usually returned to the evaporating vessel to be further concentrated for the production of a new crop of crystals, particularly if the liquid has been homogeneous.

The first crop of crystals is generally purer than subsequent ones, but may still not be sufficiently free from foreign salts and other matters, and, therefore, require to be dissolved anew and recrystallized as at first. The pure crystals are drained of their mother water by inclining the crystallizer over the evaporating vessel long enough to allow all of the fluid to run off at the spout. The crystals are then removed with a spatula and transferred to a drying frame. In the first crystallization the mass of impure crystals are drained upon a filter, and if necessary to free them from syrupy or dirty liquid, enclosed in a cloth and pressed (Fig. 276). Sometimes, especially when the crystals are not very soluble, they may be drenched while upon the filter with cold water, which carries away much soluble impurity. This solution, if valuable, may be mixed with the mother waters, and the whole after being transferred to the evaporating vessel be concentrated and again crystallized. The crop thus obtained is very impure, and requires to be drained on a cloth and pressed, and subjected to as many treatments with bone black, and renewed crystallizations, as are required to remove all color. It must be remembered, however, that bone black is only used when the coloring substance is organic, and when the characteristic color of the crystals is light, for it has no blanching action upon either organic or unorganized bodies which are naturally tinted.

In recrystallizations only as much water as is necessary to effect solution should be used, so that the mother waters may be as small in quantity as possible. The last mother waters being incapable of yielding any more crystals, may, in some processes, be reserved for other purposes; as, for instance, making new compounds. Thus, for example, the mother waters of iodide of potassium may be used to precipitate iodide of mercury from the bichloride of that metal, or of lead from the nitrate of lead, and those of chloride of barium, to obtain carbonate of baryta upon the addition of carbonate of soda.

Sometimes, however, crystallization is resorted to for the separation of one substance mixed with others which are variably soluble in the same liquid, and which do not crystallize together, but separate from the solvent when at different densities;—in this case the mother waters may contain one or more of the other components of the original substance, and hence are not useful for forming new compounds by PRECIPITATION. After the separation of each to the fullest extent by crystallization, at different temperature, the residue of liquor, unless it be of great value, may be thrown away.

As before said, gradual evaporation at a uniform temperature, and a perfect repose of the concentrated solution, give the most perfect crystals. Some solutions, however, crystallize less readily than others, and remain even days and weeks without exhibiting any sign of such tendency. In such cases, it is advisable to agitate the mass slightly or to stir it gently with a glass rod. This manipulation arouses, as it were, the molecules from their inertia, and frequently determines speedy crystallization. The resulting crystals are generally, however, confused and diminutive.

To obtain large crystals from a solution which is slow in depositing them, it is sometimes proper to add nuclei to the cold solution, these consisting of well formed large crystals of the same substance. As the solution increases its density by spontaneous evaporation, the nuclei assume a large size; but in order that their enlargement may be uniform throughout, they must be turned daily, so that the accumulation of matter may take place on all their surfaces.

This mode, as practiced in the arts, is somewhat modified. The deposition surfaces are increased by inserting in the solution strings as nuclei. When one solution is thus exhausted of its soluble matter, the strings with their surrounding crystals are transferred to as many fresh vats consecutively as are required to give the crystals the proper size. In this manner, blue vitriol, prussiate of potash, tartar emetic, and rock candy are crystallized.

When the twine loops are replaced by slender twigs or branches of wood, and the crystals are deposited in fine flakes from bulky solutions, the process is termed arborization.

Examples of arborization, where, however, crystallization is accompanied by chemical or voltaic action, are furnished by the various metallic trees, which are clusters of metallic flakes or crystals precipitated upon the surface of a dissimilar metal suspended in their solution.

*Crystallization by Chemical Reaction.*—The newly formed compounds, resulting from the chemical reaction, frequently assume the crystalline shape. Thus, for example, antimony roasted in contact with air forms crystals of antimonious acid; chlorine acting upon phosphorus produces crystals of perchloride of phosphorus. So, likewise, crystals of bicarbonate of potassa are produced when carbonic acid is passed through a concentrated solution of carbonate of potassa.

Silver displaced from its solutions by zinc forms a crystalline deposit. Sulphate of lime precipitated by alcohol from its aqueous solution also falls in crystals. Morphia, also, and other crystalline alkaloids, may in like manner be precipitated by decomposing their solutions with ammonia.

# CHAPTER XXIII.

## DESICCATION.

THE desiccation of a substance consists in the expulsion of its "moisture." The term moisture is used only in reference to that variable amount of water, and sometimes, though rarely, of other liquids which it may have absorbed, or otherwise retained in a state of mechanical union. The combined water or that of crystallization, of which many bodies are in part constituted, exists in an entirely different form, and is not usually to be expelled when the drying is preliminary to analysis. When, however, it is desired to dehydrate a body entirely, this latter water of combination is also to be dissipated.

The means of desiccation are various, and differ with the nature of the substance to be dried, its quantity, and alterability by heat and exposure.

DESICCATION OF SOLIDS.—Undecomposable salts and any substances unalterable by air or heat, may be dried by FUSION. If the amount of moisture is to be determined, the crucible and its contents should be weighed before and after the operation, the loss expressing the weight of water expelled. Those bodies, however, which will not bear the heat necessary for fusion, can be desiccated by EVAPORATION to dryness in a capsule—care being taken to renew surfaces by constant stirring.

Those saline matters which readily yield all their water by exposure may be reduced to powder or *effloresced* by subjecting them in thin layers to a draught of dry air which, if necessary, may be moderately heated. For this purpose as well as for that of drying crystals which do not effloresce, it is necessary in manufacturing laboratories to have a special apartment. This room should be smoothly plastered within, and need not be of large size. As a means of ventilation its opposite sides are pierced with small holes, which, to prevent the admission of dirt, are covered with wire gauze. The interior is fitted with trellis shelves for the support of the wooden frames, stretched over with white muslin, and upon which the substance rests between or upon, as may be required, folds of bibulous white paper. The heat is communicated by sheet iron flues proceeding from a stove placed outside of the enclosure, or by means of steam pipes fed by the generator, Fig. 10. The temperatures should range from 75 to 110° F.

This apartment is also useful for pharmaceutical purposes, for drying plants, roots, seeds, woods, &c. They may either be suspended or spread in thin layers upon frames, and repeatedly turned for the purpose of exposing fresh surfaces.

The air chamber, p. 35, may, to a limited extent, be made to replace this apartment, and in an experimental laboratory it is, together with the means mentioned in this chapter, sufficient for all purposes.

As the salts effloresced as above still retain a little water, they require to be repeatedly pressed between the folds of white paper until dampness ceases to be imparted to them. Sometimes a previous trituration is necessary to facilitate the process.

Filters containing precipitates after careful removal from the funnel and compression between the folds of bibulous paper, may be further dried in the same manner. Those, however, which contain the results of analytic experiments require more careful manipulation. For their treatment a copper-plate oven is often used. It consists (Fig. 281) of a brass soldered copper box  $7 \times 9$  inches, enveloped by a steam-



tight jacket, in the door of which are vent holes for change of air. The water, or the olive oil which is used if the substance requires a heat higher than 212° for its desiccation, is poured through the centre aperture at the top, but must not more than half fill the jacket. The lateral opening is for the reception of a thermometer, which is adjusted by means of a perforated cork, for facilitating the regulation of the temperatures.

The watch glasses, plates, or capsules in which the substances to be dried are placed, rest upon the perforated shelves in the interior.

The thermometer will indicate with precision the temperature of the bath, and care must be taken that the latter be not allowed to exceed the degree above which the body to be dried decomposes.

When for any reason it is deemed inadvisable to remove the filter from the funnel, they may both be dried together in a hot air oven, Fig. 282. The apparatus shown in the cut is a copper double or single cased cylinder, with a

movable cover, to facilitate the introduction of the substances to be dried. In its centre is a circular aperture for the reception of the thermometer by which the heat is regulated. A perforated diaphragm serves as a support for the funnels, watchglasses, capsules or other vessels, and in order to promote the evaporation, a current of air through the interior is excited by means of the circular apertures in its upper and lower circumference.

These baths are all heated over small furnaces or preferably over the gas lamp, a uniform heat being maintained by careful management of the flame.

"Fig. 283 represents an arrangement for drying substances



in a current of dry air produced by the efflux of water. For



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Fig. 282.

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this purpose a known weight of the substance is introduced

Fig. 284.



into the small bent glass tube (Fig. 284), which has also been weighed; the body of this tube is plunged into a copper water bath b, charged with a saturated solution of common salt; it is kept in its place by a cover furnished with two apertures for the arms of the drying tube; the wider arm is united by means of bent tubes and a caoutchouc con-

nector with the U-shaped tube, and containing fragments of chloride of calcium, and the narrow end is connected with a bent tube, which passes through the cork of the bottle A nearly down to its bottom. This cork must fit the bottle perfectly air-tight, and all the joints and connections of the whole apparatus must be perfect. The bottle A is filled with water, which on turning the stop-cock s flows out in a small stream, its place being supplied by the air drawn through c, and which becomes dried during its passage through the chloride of calcium, tube b. The bath is charged with water, a saturated solution of common salt, or of chloride of calcium, according to the degree of heat required, and it is kept boiling by means of a spirit or gas lamp placed underneath."

Desiccation of easily alterable Substances.—It has already been said that the power of absorbing and retaining moisture varies in different bodies. This property renders the use of those which have it in the greatest degree available for the drying of others which are deficient in it. The substances subjected to this mode of drying are mostly organic bodies and those readily alterable by heat or exposure, but which yield their moisture much below 212° F.

This method of desiccation can be conducted very well in an apparatus consisting of a large bell glass, fitting accurately upon a ground glass plate or bed. Within is a shallow saucer b, containing dry chloride of calcium, strong sulphuric acid, or other highly absorbent material, and over it a perforated glass support a, upon which rest the capsules, crucibles, beaker, watch glass, or other containing vessels. Fig. 285 exhibits the whole arrangement. The rim of the bell, as also that part of the plate which it touches, are to be greased, in order to make the joint hermetical. The material thus exposed to dry air continues to lose moisture until all has been expelled,

## DESICCATION OF EASILY ALTERABLE SUBSTANCES. 337

or until the absorbent matter has become saturated; in such case the latter must be replaced with a fresh quantity.



By substituting the bed of an air-pump for the glass disk as a support for the other parts of the apparatus, otherwise arranged exactly as above described and shown in the figure, and increasing the evaporation by exhausting the air, desiccation proceeds much more rapidly and effectually. A partial vacuum being thus produced the drying substance liberates its aqueous vapor freely, new portions being given off as soon as those which preceded them are condensed by the absorbent in the saucer, which is usually in these cases strong sulphuric acid, that agent absorbing watery vapors perhaps to a greater extent than any other. The process is thus continued until complete desiccation of the substance and saturation of the absorbent material take place, the latter being replaced by a fresh quantity when the former has not been completely dried.

If the eliminated vapors are corrosive it is advisable to modify the arrangement, so that they may be neutralized as fast as generated, otherwise the metallic surfaces of the airpump will be injured. A suitable apparatus is shown in Fig. 286. It is an inverted bell glass, fitted at its neck with a stop-cock, by which it connects with a tube containing pumice stone impregnated with acid or alkali, according to the nature of the vapors to be absorbed. The substance to be dried and the absorbent or hygroscopic body are arranged within the bell in the usual manner. The latter is then greased at its

#### DESICCATION IN VACUO.

edges, hermetically covered with a ground glass plate, and exhausted of air by a syringe coupled with the further end of the drying or chlorcalcium tube e.



By having a bed of ground glass instead of metal, and detached from the pump or syringe, and made to communicate with it by flexible lead pipe and gallows screws only when exhaustion is required, an apparatus is made, which, as represented in Fig. 285, becomes available for all the purposes of evaporation and desiccation.

Another mode of drying alterable and fixed substances in vacuo is shown by the arrangement, Fig. 287, which effects a repeated change of air. It consists of a copper cylinder

Fig. 287.



box, soldered with brass, having two apertures in its top, one, g, for the reception of a thermometer by which to regulate the temperature, and the other for a glass tube e, the recipient of the substance to be dried. This tube is connected by means of a smaller glass tube i, tightly adjusted in perforated

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corks with the chloride of calcium tube d, and thence also with the exhausting syringe b. Heat being applied to the bath\* by means of a small furnace or gas lamp a partial vacuum is then produced by several strokes of the syringe piston. In a few moments air is to be admitted through the cocks c and a, and this exhaustion and airing is to be repeated at occasional intervals, the air in its transit being deprived of all moisture by the chloride of calcium. When it is desired to replace atmospheric air by carbonic acid, hydrogen, or other gas, it may be introduced by suitable couplings with the same apparatus.

DESICCATION OF LIQUIDS.—Desiccation properly means the freeing of a body, capable of existing in a dry state, from accidental moisture. But for the sake of uniformity of description we have applied the term also to the separation, from fluids, of water, which is the ordinary source of moisture. This is usually done by the agitation with the liquid of some absorbent material, which either unites with the water, forming a stratum of different density capable of being separated by filtration or decantation; or else combines with it so firmly that the fluid which is usually more volatile can be separated by distillation.

Thus alcohol and other spirits are rectified by distillation over carbonate of potassa, chloride of calcium, or free lime, it being only necessary to stop the process as soon as the liquid comes over slowly, which indicates that all the pure spirit has passed. Agitation of ether with any of the same absorbents produces similar results.

For analytic purposes, and in minute experiments, liquids which are less volatile than water may be freed from it by exposure in open vessels under the receiver of an air-pump as described for solids in the preceding paragraphs.

DESICCATON OF GASES.—Nearly all gases in the course of elimination become involved with more or less moisture, from which it is frequently desirable to separate them previous to their application to chemical reaction. For this purpose they are passed over some highly absorbent material, such as dried chloride of calcium, quicklime, or sulphuric acid.

\* This bath is that known as *Rammelsberg's Air Bath*, which is used alone for drying substances inalterable at tolerably high temperatures.

### DESICCATION OF GASES.

The simplest arrangement for the purpose is given at Fig. 148, which exhibits a straight tube d d, containing the dried chloride of calcium, adapted at one end by means of a perforated cork with the gas generator A, and at the other, in like manner, with a disengagement tube e e. The gas in its transit through the chlorcalcium tube is relieved of its moisture. This tube varies in size from half to one inch diameter, and eight to twelve inches length, according to the quantity of gas to be desiccated. The chloride of calcium can be replaced by quicklime, potassa, or pumice stone impregnated with sulphuric acid, as the nature of the gas may require; but in either case the solid material should be in small lumps. The water formed during the process collects in this tube.

Liebig uses the drying tube of such a form as is shown at Fig. 288. It differs from the above in having a bulb, and in



being drawn out at one end to a fine tube, thus leaving but one aperture to be corked. Lumps of absorbent matter are placed in the bulb, and coarse powder of the same substance in the long part, each end of which is very loosely plugged with raw cotton to prevent the exit of particles.

For small operations the bent form, Fig. 289, is most



and operations the bent form, Fig. 263, is most convenient, as it is easily adjusted to the mouth of the bottle without the necessity of multiplying joints. The bulbs in these two latter tubes serve also as wells for the reception of the condensed vapor.

Dumas's vertical drying tube, designed for the desiccation of large quantities of very moist gas, is so constructed that the condensed vapor instead of remaining in contact with the pumice, and thus impairing its absorbent power, will be deposited in the lower part. The tube leading from the generating vessel is adapted by means of a perforated cork to a lateral tubulure at the base. The disengagement tube is similarly adapted to the top.

The selection of the drying, or hygroscopic material must, as before said, be made with a regard to the nature of the gas; thus, for example, quicklime should never for obvious reasons be used for desiccating chlorine, or other gases which combine with it chemically; for the drying of nearly all such gases an acid body may be employed, and pumice stone in lumps of about the size of half of a pea, impregnated with sulphuric acid, is very serviceable, as it presents a large extent of surface. For this purpose, however, the pumice must be freed from all the chlorides which it contains, otherwise the sulphuric acid will disengage muriatic acid possibly to the great detriment of the gas, which is undergoing drying. The best way is to pulverize and moisten it with sulphuric acid, and subject it to calcination in a crucible. When, after constant stirring it ceases to disengage acid vapors, the operation is finished.

Anhydrous phosphoric acid is also occasionally employed as a drier, but only in very nice experiments. It is mixed with clean asbestos, which occupies the same position in the tube as any of the other absorbents.

As a means of perfect desiccation it is often required to combine the absorbent powers of two different materials in one apparatus, and for this purpose the U form of drying tube is most convenient. It presents a large extent of surface in a limited space. There is, however, a disadvantage in arranging and adjusting its parts firmly together, and also in the necessity of occasionally renewing the hygroscopic substance more frequently than in the straight tubes.

Fig. 290 exhibits a proper arrangement of the U tubes for the desiccation of gas. By this mode the gas may be introduced

Fig. 290.



#### PRECIPITATION.

which are connected together by means of bent tubes, perforated corks and flexible India rubber joints. In one of their legs is placed dried chloride of calcium, and in the opposite one asbestos or lumps of pumice stone impregnated with sulphuric acid. The reservoir on the top of the gasometer being filled with water, and its pressure applied by opening the cocks, a stream of gas is gradually expelled and in its transit through the tubes is freed from its moisture by the absorbents.

# CHAPTER XXIV.

#### PRECIPITATION.

THIS process is employed for the immediate separation of a body in the solid state, both from mechanico-chemical and simple solutions. The reagent, which is used to produce the action, is termed the *precipitant* and the resulting deposit the *precipitate*.

Bodies in some instances may be precipitated unaltered, but in most cases, being the result of chemical reaction, are modified or entirely changed in their nature. Thus, for example, sulphate of lime may be precipitated from its simple aqueous solution by alcohol and the basic phosphate of magnesia and ammonia by aqua ammoniæ, they being insoluble in that liquid, the addition of which is also without chemical action upon the original solution. For like reasons the resins are precipitated from alcoholic solutions by water; and gutta-percha from solution in chloroform by ether. If, however, carbonate of soda or other soluble carbonate is substituted for the alcohol then the original combination is broken up by the action of double elective affinity, an exchange of bases taking place, and insoluble carbonate of lime precipitating instead of the unaltered sulphate as in the instance with alcohol. So also, an analogous result would ensue by virtue of simple elective affinity if soda is used instead of the carbonate, the lime then falling in a free state, having been deprived of its sulphuric acid by the caustic alkali.
The consistence of the precipitate and its form and color vary with the nature of the solutions, and the rapidity with which it is produced. These distinctive features serve as characteristics by which, in analysis, the presence of certain bodies is determined.

The precipitate is differently termed according to its appearance. It is *flocculent*, when it falls in small flakes or flocculæ, like those produced by ammonia in solutions of peroxide of iron; *pulverulent* when in fine powder and compact like the sulphates of lead or of baryta; *granular* if deposited in minute irregular molecules; *crystalline*, when it subsides in minute crystals, as the bitartrate of potassa, sulphates of silver and of lime; *curdy* when *cheesy*, like that thrown down by chloride of sodium from nitrate of silver, and *gelatinous* when of the consistence of jelly, as alumina freshly separated from alum by carbonate of potassa.

Precipitating Vessels.—The most convenient vessels, used in analysis, are the beaker glasses, Fig. 254, or

wide mouth flasks, Fig. 266, the latter being used only when the process is to be practiced upon the boiling liquid. When solutions are precipitated, especially for the purpose of collecting the precipitates, the form of the vessel may be that of the one in the drawing, Fig. 291, which ensures the subsidence of all the deposit and prevents particles from adhering to the sides. They may be of glass or blue stoneware according to the amount of liquid under process.

In chemical investigations, the test tubes, Fig. 263, are the most convenient implements. They permit the operator to use minute quantities, and they are readily heated and shaken. As a precipitate is in some instances not perceptible for some hours, especially in dilute solutions, sufficient time should be allowed to elapse before deciding upon the reaction of a preciptant upon a solution.

Directions for Precipitating.—Both the material and reagent must be in solution and separately clarified by filtration before being commingled, otherwise the suspended matters will subside with the precipitate. As heat generally promotes the reaction and the subsidence of the precipitate, the solution should, in such cases, be warmed, or even made hot, and the reagent cautiously added during continual stirring



with a glass rod so that all parts of the liquid may be brought in contact. The vessel is then set aside upon a sand bath or in a warm place until the deposition of the precipitate has left the supernatant liquor clear. A few more drops of precipitant are then added, and, if all the matter has been thrown down, they will produce neither precipitate nor cloudiness, but if a portion still remains in solution, still more of the reagent must be added. The addition of the reagent or precipitant must be gradual, for besides the waste of material and inconvenience of washing it out, an excess in certain instances redissolves the precipitate. As soon as a drop or two of reagent ceases to give cloudiness or precipitate in its descent through the supernatant liquid of the settled solution, its addition must be discontinued and the vessel placed aside, and, after sufficient repose, subjected to DECANTATION or FILTRATION to separate the solid from the liquid portion, the latter of which is also usually to be reserved in analysis or when it is of value, as it may contain other newly formed compounds dissolved in the menstruum employed.

When the precipitate about to be formed is somewhat soluble in the liquid of the original solution, the amount of that liquid must be diminished by evaporation, and the precipitation effected in a concentrated solution; for example, in the reaction of solutions of strontia with sulphuric acid or soluble sulphates.

Metals may be precipitated from their solution by other metals having a greater affinity for oxygen than is possessed by those in combination;—thus copper may be precipitated from its sulphate by iron, lead from the nitrate by zinc, and silver, arsenic and mercury from their solutions by copper. A slight acidulation of the liquid facilitates the process, and the metallic strips used as reagents must be clean and bright.

Metals are also precipitated by voltaic action, a familiar instance of which is the art of plating by GALVANISM.

# CHAPTER XXV.

## DECANTATION.-FILTRATION.

*Precipitates* which are substances deposited by any means from liquids in which they have been dissolved or chemically combined, may be separated either by decantation or filtration. The first mode is applicable to those solids which are of much greater density than the menstrua containing them, and which readily and rapidly subside forming heavy compact deposits. In delicate experiments, however, and in all cases where the liquid is turbid and deposits its suspended matter reluctantly, the latter plan is most appropriate.

Besides being a process subsequent to PRECIPITATION for the separation of the clear supernatant liquor from the subsident matter, decantation is also useful in LEVIGATION. For WASHING precipitates which require a large amount of water, or frequent renewals of the wash waters, it is much more convenient than filtration. This latter mode, however, must, as before said, be always adhered to in analyses and when the precipitate is light and apt to be disturbed during decantation.

Decantation from small vessels in nice experiments is practiced by gently inclining the vessel whether it be a capsule, as at Fig. 292, or a beaker glass, Fig. 293, and

Fig. 292.

Fig. 293.

allowing the liquid to run down in a continuous stream along a glass rod placed against its rim or edge. This ope-23

ration of *pouring* requires a degree of dexterity which is indispensable in analytic operations in order to avoid loss of material. The exact position of the rod is shown in the figures. When the pouring is completed the rod should be tilted upwards for a moment, so as to prevent the loss of adherent drops and immediately returned to the vessel, the edge of which should be slightly greased so as to effectually prevent any particle of liquid from passing over. These precautions are only necessary in the decantation and filtration of liquids, during analytic processes; so much care being unnecessary in less important manipulations, as it is of little consequence if the liquid does carry over a little of the pre-

Fig. 294.

cipitate or suffers a slight loss. If the bulk of liquid is very small, it may be removed with pipettes, Figs. 59, 60, p. 107; for larger quantities a syphon is requisite. This implement may be of glass or lead tubes, the former being cleanly and of more general application than the latter. The shapes given in the drawings refer to those of either material. The most simple form is that shown by Fig. 294, being similar to an inverted V with its opposite branches of unequal length. The long leg may be from 12 to 20 inches in length, the shorter one proportionably less. The clear diameter is from an eighth to an half inch, according to the extent of the operation.

This syphon is inserted and filled with water or any other liquid which is without

action upon that in the vessel, the mouth of the longer leg is then closed with the finger and the shorter branch introduced, mouth downwards, into the liquid to be decanted until it nearly reaches to the level of the precipitate without disturbing it. Upon removing the finger the liquid runs out in a continuous stream and may be almost wholly drawn off by slightly inclining the vessel.

The rationale of the operation is as follows:—When the short leg of the syphon is dipped into water the liquid mounts into the tube as high as the surface of that which is in the containing vessel. Now the weight of the atmosphere bears equally upon the surface of that in the vessel and in the syphon, but if the elastic force of the internal air is removed

#### DECANTATION ;---SYPHONS.

or diminished by suction with the mouth at the other end, or by having previously filled the syphon with water, the liquid runs over, and as the weight of the column of water in the long leg is greater than that in the short one, the flow will be continuous while the mouth of the short leg is immersed in liquid, for no air can enter to produce an interruption.

If the liquid is not injurious or unpleasant to the taste, the syphon may be inserted in the liquid without previous filling,—suction with the mouth at the long end drawing it over.

For the decantation of caustic liquids the syphon is fur-

nished with a lateral tube, as shown in Fig. 295, which serves as a protection to the mouth. Its application is similar to that of the one described above (p. 346); the short leg is dipped into the liquid to be decanted, the lower end closed with the finger, and suction practiced at the orifice of the supplementary tube until the air is removed and the liquid runs over and almost reaches the mouth, when the decantation goes on continuously after the withdrawal of the mouth and finger.

The annexed drawing, Fig. 296, exhibits these syphons in operation.

A length of cotton wick doubled in syphon form, and having its short end



Fig. 295.

immersed in the liquid also acts as a syphon, but is much slower in its operation.

The use of the syphon allows the separation of the liquid without disturbance of the settled matter, but as the latter still retains more or less fluid which cannot be separated in this way, it may be thrown upon a filter and in large operations even subjected to pressure in cloths, as directed at p. 320.

## FILTRATION.

The mode most commonly resorted to of separating solid substances from liquids in which they are suspended, is that of *filtration*, and it is also occasionally but rarely used for the purpose of disuniting liquids. The process consists in passing the mixture through suitable media of sufficient porosity to allow the transit of the liquid portions while they intercept any solid particles. For the separation of liquids the texture of the medium must be such that it is penetrable by or attractive of the one, but impervious to the other, of them, as it is upon this that the success of the operation depends; thus, for example, moistened paper will allow the passage of water, but not of oil.

Paper, brown muslin, linen, crash, woolen and canton flannel, felt, raw cotton, sand, asbestos, crushed quartz, bone black—each and all have their appropriate application as media, and when thus used are all styled *filters* or strainers the first title being almost exclusively applied to those of paper supported upon funnels, while the latter is limited to the other textures or bodies which are either suspended upon frames for pharmaceutical operations or deposited in proper vessels.

This process is of equal importance in chemical and pharmaceutical operations. In analysis it enables us to separate precipitates or insoluble residue from liquids, and to obtain each free from particles of the other — an indispensable condition where both are to be further acted upon for obtaining accurate results : while in ordinary operations we can by its aid free liquids from dirt and other foreign matters, and render them transparent.

FILTRATION THROUGH PAPER.-Paper is more generally

#### FILTRATION; --- THROUGH PAPER.

used, particularly in delicate experiments, than any other medium. It is advisable always to use that which is *white*, for it contains no coloring matter to deteriorate the liquid which traverses it. Moreover, it should be free from saline impurities which are soluble in acid or alkaline liquids, otherwise the accuracy of analytic results may be materially interfered with.

The laboratory should be provided with two qualities of paper, one of fine quality for nice investigations and another somewhat inferior for the less important processes. There are certain conditions requisite in both kinds. They should be unsized, yet strong, and while sufficiently porous to allow the ready passage of the liquid, compact enough in texture to retain all the solid portions.

"German filtering paper" answers very well for all general purposes, but for analytic investigations that known as "Swedish" filtering paper is the best. Being made expressly for the purpose and of purified rags, it is free from lime, copper and salts, which have to be removed from other paper by treatment with pure hydrochloric acid and repeated rinsings in distilled water before it becomes fit for such uses.

The Swedish paper is whiter and thinner than the German, and is made with great care; and leaves by incineration only  $\frac{1}{250}$ th of its weight of ashes, an important point in analyses where the amount and nature of the ashes left by the paper require to be considered.

The paper drawer should be kept always supplied with a stock of filters of all the required sizes. The use of the Swedish paper should be limited to the filtration of finely divided precipitates. The greater porosity of the German renders it more applicable for rapid filtration, and as it is much less expensive, all large filters should be formed of it.

The filters must be circular, and cut by tin patterns, which should consist of different sizes of  $2\frac{1}{4}$ , 3,  $3\frac{3}{4}$ ,  $4\frac{1}{2}$ , 6,  $7\frac{1}{2}$ , 9, and 12 inches in diameter. This mode of cutting different sized filters from one sheet of paper, is economical and saves the waste which would be occasioned by indiscriminate use of the paper, while many serious delays may be prevented by having a supply always at hand.

The ashes of the piece of Swedish filter of each size, must be determined by incinerating one and accurately weighing the residue, and engraving its weight upon the tin pattern by which it is formed. Thus, in analyses, we can know by reference to the figures the amount of fixed matter (ash) in each particular size. Kent, of New York, keeps the different sizes for sale, put up in neat boxes containing one hundred.

The supports for these circular filters, folded into conical form as hereafter directed, are *funnels* which vary in material and form according to the nature of the operation. They may be of glass, porcelain, or stone-ware. The first, free from lead, are of almost general application for analytic purposes, and the latter two for pharmaceutical. Funnels of metal are seldom required in the laboratory, a very few instances only demanding the use of lead or platinum.

The glass funnels should be made with straight sides, in-

Fig. 297.



clining to an angle of about  $60^{\circ}$ . This shape, Fig. 297, is indispensable for the smaller funnels used in analyses, as it forms in its interior a true cone, which allows the admission of a larger amount of liquid in a small space. The pint funnels, and those of still larger size, may have an inclination of ten degrees less, but if their section has not nearly the form of an equilateral triangle, the filters fit badly and work imperfectly. A slight rounding off of the angle at *a*, where the apex of the conical filter rests, greatly

promotes the filtration. The laboratory must be supplied with a series of funnels,\*

\* In addition to the above there are two other kinds of funnels used for separating liquids, which have no chemical affinity and differ in density.



Fig. 299.



ranging as follows,  $1\frac{1}{4}$ ,  $1\frac{3}{4}$ , 2,  $2\frac{1}{2}$ ,  $3\frac{1}{4}$ ,  $4\frac{1}{4}$ ,  $5\frac{1}{4}$ , and  $6\frac{1}{2}$  inches in the greatest diameter of the body *b*. Of the smaller sizes, it will be well to have duplicates or triplicates as they are the most frequently employed. The stock is not complete without one or two miniature funnels of thin glass for filtering into test tubes in qualitative investigations; and one or two of convenient size with long barrels *c*, for charging retorts and deep vessels.

Sometimes the glass funnels are ground at the rim, so as to be tightly closed by a glass disc, but being expensive they are only used in rare instances.

Funnels are sometimes made of porcelain with longitudinal

ribs in the interior of the body, as shown at Fig. 300, for preventing the adhesion of the filter to the sides in the filtration of large quantities of bulky precipitates. The object is, however, not effected by these means, for the paper sinks into the channels and adheres to the surface, and still retards the passage of the liquid. A better way will be to use the plaited filters, Fig. 308.

Funnels are also made of porcelain and more seldom of stone-ware. They are less fragile and more applicable to the filtration of very acid and corrosive liquids, and some other few purposes, than those of glass; but those of porcelain are not less costly. The form of those usually found in the market are shown in the annexed drawing. They are all glazed throughout and made very strong, and those used for transferring liquids from one vessel to another have the convenience of handles. In this respect they are preferable to the glass vessels, which by frequent rough handling are more apt to be broken. Fig. 301 exhibits the form used for acids, and Fig. 302 the same funnel ribbed in its interior. Figs. 303 and 304 present the less convenient globular shape. Those shown at Figs. 305

They are fitted with stop-cocks in their barrels, as shown in Figs. 298 and 299; and one is stoppered also at the top to prevent evaporation when volatile liquids are under process.

The mixed liquids of oil and water, or ether and water, for instance, are poured in the mouth, and after sufficient repose for the deposition of the heavier of the two, it can be drawn off by opening the stop-cock which may be immediately closed as soon as all has passed. The lighter liquid which is thus retained may afterwards be transferred in the same way to another bottle.

Fig. 300.



and 306, cullendered at the base, are the most convenient of all, being very useful for draining crystals, for the filtration of viscous solutions through cloth filters, and for small operations of lixiviation.

Filters Folded and introduced into Funnels.—Two kinds of filters are generally employed, the plain, Fig. 307, and the plaited, Fig. 308. The former are used in analyses and

Fig. 307.

Fig. 308.



whenever the suspended or precipitated matters of a liquid are to be preserved. It is almost impossible to entirely remove the solid matter from the folds of a plaited filter, consequently such are chiefly applicable for the filtration of bulky precipitates from large quantities of liquid. This mode of folding a filter prevents its close adhesion to the glass, and greatly expedites the process by increasing the surface, and by allowing a bubble of air to ascend in the fold every time that a drop of liquid descends from the filter.

The plain filters are folded as follows:---

"When a filtration is to be performed, one of these circular papers of the proper size is selected (Fig. 309), and then doubled over one of its diameters (a b, Figs. 309 and 310), and then over the radius (c e, Figs. 310 and 311) perpendicular to the first diameter, so as to form a quadrant. One



of the folds is then opened, forming a hollow cone, as represented in Fig. 313, which will fit accurately in the funnel, if the sides of the latter form an angle of  $60^{\circ}$ . If the angle be greater or smaller, it is necessary to double the filter the second time over another radius (*c f*, Figs. 309 and 312), not



perpendicular to the first diameter, and then open the large or small fold (*a c f*, or *b c f*, Fig. 312), according to the angle of the funnel, and this repeated until a coincidence of the filter with the inside of the funnel is effected."



To form the plaited filter, take a square of paper and fold it diagonally, as in Fig. 314; turn A upon B to obtain the crease E and open it; then double A upon E in the same direction, to make the plait F, and double the plait A back upon F, so as to form the crease G, and holding this plait between the fingers make the fold between F and D. Divide the spaces between E B and B D in the same manner.

The filters, as above made, after having their folds opened, as at Figs. 307 and 308, are placed in the funnels and so adjusted as to fit nicely to the sides. In order to secure an uninterrupted flow of the liquid, and to prevent the breaking of the filter, the apex must not extend too far into the barrel of the funnel. Moreover, the filter should be a little smaller than the funnel, for if it reaches to the rim, evaporation of the liquid ensues from the edges, and thus, in analysis, may be a source of error.

The proper position of the plain filter in the funnel is shown at Fig. 316, and that of a plaited one at Fig. 315.

In using large funnels the filter may be supported by a plug of raw cotton placed in the barrel at its junction with the body.

Fig. 315.







The usual support for funnels is the convenient portable stand, Fig. 316. It consists of a wooden upright b, screwed into a wooden bed plate. The arm a, which it carries, has a circular aperture sloping inwardly and downwards, which supports the funnel steadily in its place. The screw c allows the elevation or depression of this arm at will, as the height of the receiving vessel beneath may require.

When the funnel is used for transferring or filtering liquids into narrow-mouthed vessels, its barrel may be supported by their neck; but in order to secure a free passage of air it should be fluted externally, or else have a chip or two placed between it and the inner sides of the neck, otherwise the confined air will retard the process, and possibly force the filtered liquid, with a hissing sound, up and over the sides and mouth of the bottle.

After having adjusted the filter to the funnel, the latter is placed in the stand, so that its barrel may rest against the inner wall of the receiving vessel beneath. This position allows the falling fluid to trickle quietly down the sides, and prevents the splashing which would occur if it fell directly upon the surface of the liquid, and also obviates the necessity of sinking the barrel far into the receiver.

The filtering apparatus having been thus arranged, the filter is to be moistened with distilled water from the bottle, (Fig. 38,) or when the nature of the process requires, with a portion of the solvent liquid, and the excess allowed to trickle through, rather than be emptied out by inverting the funnel. This previous soaking of the filter greatly facilitates the operation, for dry paper absorbs water directly, and in the case of a turbid solution, while becoming more impervious to the suspended particles than it would be if the liquid which contains them were allowed to penetrate at once into the filter, it gives also a more ready passage to the clear fluid. The edges of the containing vessel are now to be slightly greased in one spot, so that in pouring there may be no adhesion of drops or trickling over the sides. It is then grasped by the right hand and brought over the funnel, while the left hand holds the glass rod at a right angle against the edge of the glass as shown in Fig. 317. The end of this rod should merely reach the filter without touching it, for fear of abrasion; and the liquid should be allowed to flow down its length in a gentle stream at first against the sides, and as the precipitate accumulates it may be allowed to fall in the centre, as there is then less risk of splashing. The filter should never be entirely filled, and as it often requires many pourings to pass

#### FILTRATION :--- POURING.

the whole of the liquid, great care must be taken in returning the rod to the vessel that nothing be lost. The last particles



may be rinsed from the vessel and rod by the jet of the spritz bottle A,\* (Fig. 321,) by inclining both to the positions shown

\* The spritz, or washing bottle, consists of a twelve ounce vial, to the mouth



of which is adapted, by means of a perforated cork, a glass tube, drawn out at its upper end as shown in Fig. 318, which represents at the same time its exact dimensions. The bottle is rather more than half filled with water, and by blowing into it through the tube the air is compressed, and when the bottle is quickly inverted it forces out the water through the orifice in a strong jet, which



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in the drawing below. If any remaining particles still obstinately adhere to the sides of the glass, or of the rod, they



must be loosened by the feather end of a goose quill, and then washed out as before by the jet of the spritz. When all the liquid has passed through, the precipitate must be washed down from the sides of the filter by the force of the jet of water from the spritz.

In dusty apartments, both funnel and receiving vessels should be kept covered by circular or square pieces of window glass. The one over the receiver should have an opening in the side for the passage of the barrel or tube of the funnel.

The receivers are most generally beaker glasses, but capsules, flasks, and narrow necked bottles are all made use of. The above precautions refer especially to

filtrations in analytic operations. In larger operations the manipulation is not, necessarily, so strict, and when the dimensions of the containing vessel will not admit of convenient handling its contents may be con-



veyed to the filter by ladlesfull in the small porcelain dipper.

may be directed to any desired point. The bottle complete is exhibited at Fig. 319. For washing out beaker glasses, or other deep vessels, a curved jet is more convenient, and is seen at A, Fig. 321.

When hot water is required the bottle should be of copper, of at least a pint capacity, and of the form presented by Fig. 320. It is heated as directed at p. 231, Fig. 185, and to prevent burning of the hand, is fitted with a non-conducting handle. Upon inversion of the bottle the water is driven through the tube d in a strong jet by the elastic force of the confined vapor.

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Fig. 322. The ladle, during the intervals of the transfers, must rest in a plate and not be placed anywhere in the dust.

In order to expedite the process, the liquid, as a general rule, should be allowed sufficient repose previous to filtration, to deposit if possible all its suspended matter, and the clear supernatant portion should be passed through first. The subsident matter being added last, is filtered, as it were, alone, and offers no impediment by obstructing the pores of the filter to the passage of the liquid portion, as it would if mixed with it. As an exception to this rule, certain precipitates which are curdy, gelatinous, flocculent or crystalline, may be filtered immediately after their formation. As warmth usually expedites the process, nearly all liquids, when circumstances permit, should be filtered whilst hot.\*

• It has already been said that heat promotes filtration; it is even necessary for saturated solutions, which are liable to deposit crystals upon the least diminution of their temperatures. Below are drawings of two kinds of apparatus, convenient for keeping liquids warm during the operation. Fig. 323 represents that known as Professor Hare's filter bath, and consists of an oval copper jacket, flat at top and bottom, with two conical apertures through its body. The cone, with its expanded part directed downwards, is a sort of chimney, under which a spirit lamp is placed to heat the water in the bath, and the other is a bed for the funnel. To prevent ignition of the vapors when inflammable liquids are under process, there is a partition beneath.



The other drawing, Fig. 324, also shows an apparatus in which there is a metallic casing for the support of a funnel. The neck through which the funnel passes is closed with a perforated cork, and the contained water is heated as shown in the figure.

Both of these implements are fitted with covers.

For filtrations of heavy precipitates, or a large amount of liquid, it is advisable to use the filter doubled or even trebled, as it will be thus enabled to resist a very heavy weight. This precaution is necessary also when the liquid runs through a single paper turbid. When only the first runnings are turbid, a single filter will answer for small experiments, but the liquid must be repassed through the same medium.

FILTRATION THROUGH CLOTHS.—In large operations, or when the solid matter to be separated is too heavy, or would corrode or clog the pores of paper, the latter is replaced by cloth. The kinds of cloth vary, and each of those already mentioned has its appropriate application. The texture of the medium must be adapted to the consistence of the liquid; for example, flannel or felt may be used for filtering mucilaginous, saccharine and slightly acidulous solutions; twilled cotton or canton flannel for oils; linen and muslin for tinctures, vegetable juices and dilute alkaline leys. Sieves of bolting cloth are occasionally used for filtering liquids from very fine or flocculent matters.

Filters made of the materials above mentioned, and which take the name of strainers, instead of being used like those of paper, are stretched upon square frames formed of four pieces of lath, as shown at Fig. 325. These frames, of which



there should be several sizes, must be strongly jointed, and should have inserted upon their upper surfaces a number of rectangular hooks, similar to those used in the drying lofts of calico factories for hanging up the printed goods. The cloth, of whatever kind, being cut into a square of size proportioned to that of the frame, is stretched over it somewhat loosely, and retained in position by hitching its margin on these tacks or

#### FILTRATION THROUGH CLOTHS.

hooks. This mode is far preferable to that of nailing the cloth down with flat headed tacks, for besides the injury of material, there is less convenience in removing it after the filtration for pressure, or for replacing it with another when it is required. The support for these strainers is an upright stand, Fig. 326, the interval between the legs of which is sufficient to allow the free entrance of the receiving vessel.



When the cloths are made into conical bags, as is very often the case, and not without advantage, they are to be suspended by loops to a transverse beam, as shown in Fig. 327. These bags allow the convenience of using narrow mouthed receivers, as the liquid trickles through in a stream from the most depending parts.

The texture of the straining cloth must be porous, but sufficiently compact to prevent the passage of any solid particles. It is far more convenient than paper for coarse filtration of decoctions, tinctures, oils, syrups, and for separating liquids from solid organic matters. In most instances the cloth or bag may be renovated by washing, and thus be rendered fit for other operations.

Before stretching the cloth upon the frame, or suspending the bags, they should be first moistened with water, or if necessary with a portion of the solvent liquor in order to swell the fibres and contract the meshes. The liquid should be then

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## FILTRATION THROUGH PULVERULENT MATTER. 361

introduced gradually without spilling. For this purpose a tinned, copper, or porcelain ladle, Fig. 328, with a wooden



handle, is very convenient. A very excellent substitute is a dipper, made from a cocoa-nut shell, and sold in any of the furnishing shops. Additions of liquid should be made until the filter is nearly full, and it should be kept at the same level by renewing it as fast as it runs through. If the first runnings are turbid, they should be returned to the filter, and if they continue murky, repassed through a fresh cloth.

After all the liquid has passed through in this way, the cloth or bag is to be unhooked, carried to a table, securely folded, and enveloped in a wrapper, and subjected to pressure as directed at p. 320, for the expulsion of the retained portion of liquid. The precipitate thus pressed, when an object of value, is to be cut up with a spatula and spread on frames for DESICCATION.

The cloths are then to be immediately rinsed and cleaned in water without soap, dried and placed away for service at another time.

FILTRATION THROUGH PULVERULENT MATTER.—Crushed quartz, clean white sand, asbestos, bone black, and charcoal are the materials generally used as media. The two latter act both as filtering and purifying agents, as the liquid becomes not only clarified in its passage, but freed from coloring and putrescent matters, if any exist in it. The others are used for the filtration of very acid or corrosive liquids, which would be destructive of paper or cloth, and partially solvent of bone black.

All of these substances may be used in funnels, a thin stratum being placed in the bottom of the body, and prevented from escaping through the barrel by a loose cotton plug in the neck.

A funnel plugged in this manner, even without the stratum of pulverulent medium, answers an excellent purpose for the filtration of liquids which pass through freely, and whose suspended matter is in coarse particles.

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It will of course be remembered that the use of these media is only practicable when the liquid is the sole object of value, for it would be impossible to prevent at least the partial admixture of the suspended matter with the secenning agent.

The asbestos, sand, and charcoal should first be treated with muriatic acid to remove soluble matters, and then thoroughly rinsed with fresh water to remove all traces of acid previous to their employment as filtering means. Freshly prepared and finely powdered charcoal, by its absorbent power, deprives most liquors of their fetor and organic coloring matter; bone black has the same effect, but in a much less degree. These two are the best substances for separating impurities from syrups and aqueous liquids.

The filtering substance should always, before being used, be moistened throughout, as in displacement, with clean fluid, or, as is proper in many cases, with the pure liquid which is the solvent of the various substances in the fluid which is to be depurated. Thus the substance in the funnel may be made to imbibe water before the filtration through it of a syrup, and alcohol or ether before the passage of tinctures or ethereal solutions.

Where a natural repugnance exists between the particles of the fluid and of the filter, as is the case with finely divided charcoal of any kind and water—or light aqueous solutions the solid must be made to absorb the first parts of the fluid by thorough agitation and trituration with it, and then be allowed to separate, previous to its employment, by deposition. In other cases, the pressure of a high column of fluid upon the substance must be allowed to compel the necessary union of surfaces.

Filtration by DISPLACEMENT, to which the above mode is in some respects similar, has already been fully described at p. 314.

FILTRATION OF VOLATILE LIQUIDS.—Donovan has contrived an apparatus for filtering liquids which are vaporizable or alterable by exposure to air. It is identical in principle and construction with the displacer, Fig. 275, and is very useful for filtering alcoholic, ethereal, or ammoniacal and alterable caustic liquids.

The modification of Riouffe is more convenient than the original apparatus, as it allows the use of an ordinary funnel with a cover. It is represented by Fig. 329, and consists of a glass

#### WASHING.

bottle A, with two necks; into one of which enters the barrel of the funnel. The neck of this funnel is loosely closed with



a plug of raw cotton, and the liquid is introduced through the s tube without uncovering or disturbing the apparatus. As the liquid filters through, the column of air displaced, finds a vent through the narrow tube a, adjusted in position by means of perforated corks. The stop-cock K allows the withdrawal of the filtrate at pleasure.

# CHAPTER XXVI.

## WASHING.

IN all precipitations, the powder thrown down becomes involved with more or less of the original liquid from which it has been deposited. As these liquid portions are impurities, they must be separated, and in many large operations, and when the precipitate is bulky, we effect their removal by repeated washing and DECANTATION; but when the powder is light, and in all cases where accuracy in estimating results is required, the purification is conducted by pouring continued streams of water or other fluid through the substance contained in the filter.

Washing by decantation is usually practiced by diffusing the precipitate in a large quantity of cold or hot water, or other suitable liquid, as circumstances may require or admit; stirring well, and after sufficient repose for settling, decanting the clear supernatant solution. A repetition of additions of fresh water, and subsequent decantations after repose, will entirely remove all soluble matter and free the precipitate from impurity.

In analyses, the precipitate is most generally washed upon the filter by projecting water from the spritz bottle A, Fig. 321,



the jet of which, by its force, at the same time loosens that portion adhering to the sides, and concentrates it all at the bottom, when a larger amount of washing liquid may be added from the bottle, Fig. 38. To give force to the issuing jet, as is sometimes necessary in detaching particles from the filter, the tubes of the spritz may be as shown in Figs. 330, 331. The compression of the air in the interior by blowing in the tube a produces a jet through the lateral one b, drawn out to a small orifice. This form of spritz is much more convenient for large filters than the smaller one, Fig. 319, either, however, allowing the direction of the stream to any desired part of the filter.

The above mentioned bottle is flat bottomed, and of thin glass, so as to answer for the use of boiling liquid.

The copper flask, Fig. 186, with tubes fitted to its mouth as above described, is, however, far more convenient, and less liable to receive injury.

The precipitate being in this manner kept constantly mingled with liquid, is soon freed from its soluble matter. The latter fact is known when a drop of the wash-liquor, which

## EDULCORATION; - WASHING BOTTLES.

has passed through, leaves no stain upon a silver or platinum spatula heated over the spirit lamp.



There are many precipitates which require protracted washing, or *edulcoration*, as it is sometimes termed, in order to cleanse them thoroughly, and the bottle for the purpose is so



Fig. 333.



constructed as to be self-operating in a measure, this mode being a great saving of time and labor to the operator. Fig. 332 represents the whole arrangement, which is so contrived that by a suitably constructed tube b, adapted by means of a perforated cork to the flask or bottle a, the water therein contained flows out very gradually, and in quantity proportional to its passage through the filter. The tube is that known as Gmelin's. It is well replaced by two separate tubes, which can be readily formed over the blow-pipe flame by the operator himself; the modified implement is shown at Figs. 333, 335.

Below are the two forms of washing tubes, both acting upon the same principle. Fig. 334 represents the one devised by Berzelius, and Fig. 335 that of Gmelin's.

The mode of washing by these bottles is very convenient. They are nearly filled with water, and inverted over the funnel in such a position that the part e extends below the surface of the liquid and no further. The flow continues in a constant current without further attention until the surface of water in the filter rises towards the line ef, Fig. 335, and diminishes the pressing column, when capillarity is in excess and no more water flows. But as the water slowly percolates through the filter, the column is increased, and the water again flows. This alternate action is continued until the bottle is emptied.

The great convenience of this arrangement is that a filter may be washed during the absence or inattention of the operator.

If the precipitate is soluble in water, it must be washed with alcohol, ether, or other liquid which is without action upon it.

When the bottle filled with water is inverted, as in Fig. 332, there will be no efflux of water from the small opening c, Fig. 335, so long as this point is at a certain distance below the curve of the syphon; but if the moistened finger, or other body, be held to the point c, water will flow freely from it, and air bubbles will ascend through i b to supply its place. If the tubes and opening were of large size, the water would flow out with touching the end of the tube, but being of small diameter, and the end c being drawn out finely, the efflux of water is opposed by capillary attraction. The column of water between e f and g h is the force tending to overcome the capillary resistance. If this column be lengthened by drawing e d

### BLOWPIPE MANIPULATION.

further through the cork, then the water will flow out of e spontaneously. If c d be pushed in just so far that the water



does not flow spontaneously, then the capillary resistance slightly predominates. Then if a substance to which water adheres by adhesive attraction, be applied to the end of c, it will make the water flow so long as it is held there, because the adhesive attraction of the touching overcomes the capillary action. If c d be thrust still further into the cork, then capillary action predominates so greatly that no adhesive force can counteract it, and the water will consequently not flow out. There is, therefore, a particular medium position for the point c, where it will act as desired.

## CHAPTER XXVII.

## BLOWPIPE MANIPULATION.

MANY substances come under the observation of the chemist, of the nature of which the physical properties furnish but little indication; and as a preliminary examination called *Qualita*- tive Analysis, should in such cases be made to precede the Quantitative Analysis,—in order that from a knowledge of the constitution of the body, a mode of effecting the latter process, or the determination of the amount of its constituents, may be devised—it becomes very important to be provided with a means of simple and rapid examination, and one of general application.

Such means are presented by the *mouth blowpipe*, which is simple in construction, cheap, portable, and which not only fyrnishes to those who are at a distance from, or unfurnished with chemical apparatus, the power of determining the character of minerals and other bodies, but when employed as an adjuvant to other methods, enables us frequently to appreciate with exactness and ease the most minute quantities of simple bodies, or the ingredients of those which are complex.

Even in analyses in the humid way, we are often obliged to resort to it, in order to be enabled to ascertain the existence of a substance, the presence of which in solution cannot be determined by any tests.

The blowpipe is employed for the purpose of forcing a fine stream of atmospheric air through the flame of a candle or lamp, so that the continuous current or blast produced, shall impel in a proper direction, the flame, and furnish to the partially burnt particles of carbon of which it in a measure consists, enough oxygen to cause vivid combustion and great heat.

The simplest form of a blowpipe, and the one originally adopt-

ed, is that used by gas-fitters, jewelers, and others, for the purpose of soldering, &c. It is represented in Fig. 336, and consists of a metallic tube, usually made of brass, somewhat curved at a short distance from its tapering extremity. The bore terminates in a very small perforation, with a rounded margin.

This instrument is used by propelling a rapid and steady blast of air through the tube from the mouth, by the action of the muscles of the cheeks, and by directing this blast against the side of the flame. The blowpipe of this form is still preferred by artificers for

operations in which little blowing is needed; but when the process is of some duration, the moisture of the breath gradually condenses in the tube, and impedes the blast, or else the latter forces some of the aqueous matter through the flame upon the substance which is under examination, thus consti-

Fig. 336.

tuting serious objections to its employment for analytical purposes.

Of all the modifications of this instrument, that of the celebrated Gahn is by far the best, and to it almost universal preference is given. Before proceeding to speak of it, however, it may be well to refer to the ingenious contrivance of Dr. Wollaston. It consists of three pieces, a, b, and c, Fig. 337. The small end of the tube a fits in the large end of b. The latter is closed at the other and narrower extremity, and a short distance from it, a small hole, d, is pierced transversely through the tube.



The different parts of the instrument are represented to better advantage in Fig. 338. The smallest piece, c, which has its short end closed, is slid over the top of b, by means of the oblique hole c, in such a manner that the small hole d will

### GAHN'S BLOWPIPE.

communicate with the fine conduit in the narrow end of the former piece. This instrument possesses the great advantages of being compact and portable, for when properly constructed, all its pieces will exactly fit in each other, and when closed the whole is scarcely larger than a pencil case. It is shown thus packed up in the figure. The objections to it are that it contains no air chamber, or suitable reservoir for retaining the condensed moisture, and that the direction given to the blast in consequence of the angle formed by the piece c with b, is such as to prevent the operator from properly seeing the substance of which he is investigating the properties.

Gahn's admirable instrument shown in Fig. 339, combines the advantages of all former inventions.

Fig. 339.

A long and slightly conical tube fits in a cylindrical chamber which is one inch in length, and half an inch in lateral diameter, and which is designed to condense and retain the moisture. In the side of this cylinder is inserted another and shorter tube, which makes a right angle with the large one, and which is considerably less in size. That end of it which is introduced

Fig. 340. 

This metal is preferred on account of its infusibility, and of the ease with which a common impediment to the operation —the clogging of the mouth of the tube with finely divided soot—can be removed, when the extremity is made of it. All that is necessary to get rid of this deposit, is to subject the clogged tip of the tube to the action of a high heat, produced by the use of the same blowpipe, and which burns off the carbon. Tips made of silver answer a very good purpose, and are often used; but they are apt to become brittle and crystalline in texture upon cooling, after exposure to a high red heat.

When moisture collects in the chamber of this instrument, it can be expelled by simply disconnecting the joints, blowing forcibly through the tube, and by the application of a dry cloth.

Mitscherlich has made an improvement upon Gahn's blowpipe, which renders it more portable. He reduced the size of the chamber and fastened it permanently to the long tube. This tube is made to unscrew in the middle, so that the small tube c, with its platinum jet D, can be slid into the part connected with the chamber, and the other half A, can be fitted upon B, so that the whole makes an instrument little inferior in portability to Wollaston's, as shown at F. The little nozzles adjusted upon these instruments\* can, in a measure, be dispensed with if care is taken to remove the blowpipe from the flame the moment the blowing is suspended. When the small orifice becomes filled with soot it can be reopened by introducing the point of a needle, which has been held for a short time in the flame. If this latter precaution is not attended to, the point is apt to snap off, and sometimes great



difficulty is experienced in removing it, and risk incurred of injuring the instrument.

\* They are preferred for cheapness, and should be silvered or platinized.

Dr. Black's is the most cheap form of metallic blowpipe, and is shown in Fig. 342. It is a conical tube of japanned tinned iron plate, closed at the wide extremity; near which, upon the side, is adapted a brass tube with a nozzle of proper size.

Silver and tinned iron are the proper materials for the construction of blowpipes. Copper, brass, and German silver are employed, but being exposed both to heat and moisture, they oxidize easily, and give an unpleasant odor to the hands and brassy taste to the mouth.

Necessity may compel the student to make a blowpipe of

Fig. 332.

his own construction. In such a case, a common clay tobacco pipe may be converted into one by closing the bowl with a cork, in which is fitted a glass tube, with one end drawn out to a small orifice. It is economical and convenient, and considering the fragile material of the exit tube, answers the purpose admirably.

One of the best blowpipes we have ever seen employed was constructed in this way, except that the small tube was made by filling the end of the bore of a broken tobacco pipe, firmly with common putty, and then piercing the plug thus made with a pin or needle, and allowing it to dry.

This expedient succeeds very well, and although the instrument is clumsy in appearance, the extremity is not fusible like glass, and with a little skill the whole may be so fashioned as to make a most serviceable apparatus.

Entire blowpipes are also readily made from glass tubes. The material is cheap, and the requisite form can be easily given them; but notwithstanding these advantages, their use is very limited, in consequence of their brittleness, and the ease with which the point of the small tube fuses.

The blast from the blowpipe is directed upon the flame of a wax or tallow candle with a cotton wick, or that furnished by coal gas or an oil lamp. If a candle is employed, Bergmann directs that the wick be inclined to one side towards the object, i. e. in the direction in which the flame is to be impelled. Besides not always giving sufficient heat, candles have the disadvantage of consuming too rapidly, since the wax or tallow readily melts from the heat which is radiated from the substance in the flame. The wick also requires frequent

## BLOWPIPE LAMP AND APPLIANCES.

trimming. The lamp recommended by Berzelius, with the improvements of Harkort, gives the best flame for these experiments, and has a very convenient form. It is seen in Fig. 344. It is made of brass,\* has a length of  $4\frac{1}{2}$  inches, is



of a slightly conical shape, and is an inch in diameter at the lower end, which is furnished with a screw to adjust it to the brass rod, which is fastened to metallic cross pieces, so as to support it in its perpendicular position. The screw enables the operator to raise or lower the lamp to suit his convenience. On the upper side of the lamp, at one end, is an opening for pouring in the oil, closed by a screw cap with a leather washer. Near the other end is the wick holder, a piece of tinned iron of a rectangular form, which is inserted in a brass screw permanently connected with the lamp. The direction of the wick holder is parallel with the lamp. When not in use a screw cap with a washer is adapted to the corresponding screw, and surrounds the wick, closing it hermetically, so as to prevent the escape of oil in transportation. The object of the obliquity of the front of the lamp is to permit a considerable deflection of the flame when it is desired to bring the assay nearer to the source of heat, as is sometimes necessary. Fig. 345 represents the cap covering the wick, and gives a view of the position of the cross pieces forming the support of the lamp.

\* Tin plate may be substituted.

The rod can be unscrewed in the middle, the cross pieces disunited, and all the parts separated, so that the whole can be packed away and made to occupy very little space.

This lamp is fed with pure olive or refined rape oil, which

Fig. 345.



is decidedly the best. For the stationary blowpipe table in the Laboratory, coal gas is used, and is supplied by a leaden pipe with a nozzle of brass, similar in form to the wick holder of the lamp; it does not furnish the same amount of carbon for combustion as the above named liquids, but is preferred on account of its cleanliness. The triangle, Fig. 344, with the bars, attached to the arm which moves laterally, by a screw capable of being moved upon the upright, is for the support of vessels, such as small crucibles, over the flame. Different sized crucibles can be made to fit in this triangle by

removing one or more of the bars.

In some cases the common spirit lamp, Fig. 115, before described, is used to furnish the flame. It answers particularly well for the heating of glass tubes when it is desirable to avoid a deposit of soot upon the surface.

The Flame.-The flame of a common candle or oil lamp con-

Fig. 346.



These different appearances are accounted for in the following way. The wick—which, after its combustion has melted the upper portion of the

fatty matter, is merely the agent for the imbibition and passage upwards by capillary attraction, of the fluid combustible—gives off at its ignited extremity the inflammable gases and possibly some carbon, into which the fat has been resolved. These gases, not meeting with a suffi-

cient supply of oxygen, rise and form the central dark cone; upon the outer surface of which, however, the hydrogen contained in them may be supposed first to combine with the oxygen of the air, to the production of an intense heat, by exposure to which the carbon which had been unconsumed is heated to whiteness; the latter thus forming the brilliant luminous portion of the flame by its ignition, and more or less complete union with oxygen. The external envelope of faintly luminous matter is probably owing to the complete combustion in contact with air, of those portions of gaseous matter which had not been previously burnt. This part, considered in reference to its whole surface, is the hottest portion of the flame, but the maximum of heat is given off at the level of ff in the Fig., and from that part it gradually decreases to the apex and the base. The combustion of a little carbonic oxide, and possibly some carburetted hydrogen, gives rise to the blue portion of the flame seen at a, b. This is the coolest part of the flame.

A stream of air projected into the flame makes it present a very different appearance. Before the nozzle of the blowpipe, is formed a blue, well defined, long and slender cone, which is similar in appearance to that in Fig. 346. The hottest point in the flame thus excited is just before the point of the cone. Exterior to it is a yellowish-brown flame a c, somewhat luminous, but undetermined in outline. It is in this flame a little beyond

Fig. 347.

the point of the blue cone, that reduction is effected. For oxidation we must remove the body from the outer flame just so far as the temperature is consistent with the object in view, i. e. at such a distance in advance of the inner flame, as is best calculated to oxidize. But as metals differ in their affinity for oxygen, this point can only be ascertained by practice.

The former is called the reducing flame, because in consequence of an excess at its extremity, of burning carbonaceous matter, which greatly absorbs oxygen, the oxide of a metal, if employed, is deprived of its oxygen, or in other words, is reduced to the metallic state. One object of the blow-pipe is to supply oxygen, which is always contained in air expelled from the lungs, so as to burn off the hydrogen of the flame and to set free the carbon and carbonic oxide in order to reduce the reducible body exposed to its action. It is also the co-operation of some unconsumed carburetted hydrogen that assists in the reduction.

The Manner of Holding the Blowpipe.—The cut, Fig. 348, represents somewhat imperfectly the proper mode of



holding the blowpipe and support, and of directing the flame upon the object on the latter. The former is held like a pen between the thumb and first two fingers of the right hand, and its mouth-piece is inserted between the lips. The support is held in a convenient position by the left hand, and both arms should be so fixed upon the elbows, or otherwise, without reference to a particular fashion of holding them, as to ensure that the object be kept constantly in one place under the continuous blast from the blowpipe.

The Blast.—A uniform current of air is expelled through the tube from the lips, by making the mouth and lungs act upon the same principle as the ordinary table blowpipe, which has been before fully described. The lungs acting like the piston, force by their alternate contraction and dilatation, an intermitting current of air into the cavity of the mouth; which being analogous to the condensing box of the large blowpipe, by the constant pressure and elasticity of its muscular walls, converts the alternating into a continuous blast. The be-

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## BLOWPIPE MANIPULATIONS :--- SUPPORTS.

ginner finds great difficulty in properly regulating this part of the process, and some never acquire the necessary tact. It is an accomplishment which experience and practical instruction alone can give, and which no description can impart. The blast is first forced through the tube by filling the mouth with air, expanding the cheeks, and then keeping up a constant and forcible pressure with the muscles forming their parietes, at the same time that respiration through the nose is allowed to go on calmly and uniformly as usual. But as the current constituting the jet is required to be uniform, it must be prevented from sharing in the alternating impulse communicated from the lungs, by a valve-like closure of the opening of the mouth into the throat; which closure becomes, with a little practice, an instinctive act. When the mouth is nearly emptied of its air, this communication is temporarily reopened without any intermission of the blast, the cavity is refilled, and the communication again closed until the next occasion for its opening.

Supports.—The substance under examination, must be allowed to rest firmly upon a support, which should be such as will not fuse under a high heat, combine chemically with the fused body, or prevent its complete heating by rapid conduction. The supports in most common use are charcoal, and platinum either in the state of wire or foil.

Charcoal makes, for many operations, an excellent support, especially that kind of it which is made from well-grown pine wood or the branches of the willow. It should be well charred, and that which snaps or smokes in the fire should be rejected. It is desirable that it should be as free as possible from ashes, which nearly always contain a trace of iron and manganese; therefore dense and compact woods should not be used, as they give much ashes, and often contain a considerable amount of those oxides, which by uniting with the fluxes employed, would give incorrect results. Straight pieces, free from knots, should be sawn in the direction of the fibres, into oblong supports of the proper size. The assay is placed in a shallow concavity made near one end of such a support by the borer or point of a knife; and upon this substance, so prepared, oxidation, reduction and fusion are chiefly performed.

Sometimes the reducing property of charcoal, and the rapidity with which it is dissipated into carbonic acid, inter-

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fere with the result. In such cases platinum in the form of foil or wire is used. A narrow strip  $\hat{3}$  inches long and  $\frac{1}{2}$  inch broad is often advantageously employed for oxidation. The substance which is to be oxidized, is placed on it near one end, and heat is applied by the blowpipe flame upon its under side. Its conducting power is so inconsiderable that the other end may be held between the fingers without inconvenience. Platinum cannot in general be used for reduction, as it forms fusible alloys with some of the metals; nor should sulphurets, arseniurets, &c., be heated in contact with it. When the strip is too short to be held between the fingers, it can be inserted in a piece of wood or charcoal, or be held between the points of the pincette. A small spoon of the same metal is sometimes made use of; but in the majority of cases the wire may be substituted for any other means.

When platinum wire is used, it should be moderately thin,

Fig. 349.

and have a length of about  $2\frac{1}{2}$  inches, and should be bent into a hook at one end, which serves as the support for the assay. This part is either heated for a moment in the flame or moistened by the tongue, and dipped into the flux, whereby a small quantity becomes attached, which, when fused to a transparent bead by the blowpipe flame, becomes firmly fixed in its bed and occupies the space within the curve. The side of the bead is then moistened and a little of the assay is made to adhere to it.

Both are now fused together, and the appearance of the bead held in one or the other part of the flame, in reference to opacity, color and other characteristics, can be distinctly seen from all sides, and in this way are colorations of the bead by metallic oxides particularly to be distinguished. The only objection to this hooked wire, occurs in the case of the use of *fusible flux*, which is apt to fall through it. Two or three additional turns of the hook will generally make a bed sufficiently close to prevent this salt from running through when fused.

The bead can be detached from the wire, when cold, by a slight blow with the hammer. If, after its removal, the wire is not left perfectly clean, a bead of soda may be fused upon it, and afterwards dissolved out, when it takes the impurities with it. In extensive investigations by means of the blowpipe a number of these wires should be provided.
#### DETECTION OF VOLATILE SUBSTANCES.

Detection of Volatile Substances by means of the Blowpipe.—When volatile substances or gaseous products are to be tested by means of this instrument, the body to be examined is usually exposed to heat in an open glass tube, which may be from two to four inches in length, and from the twelfth to the third of an inch in diameter. The body is placed near to one end and the blast is directed upon it, while the tube is inclined more or less in proportion to the current of air, which is required to be passed through it. By such means, disengaged vapors may be sometimes recognized as they emerge from the upper end, and volatile matters condensed upon a part of the tube.

It is often necessary to deposit the substance in the angle of a curved tube so as to prevent it from falling out. A tube so bent is shown at b, Fig. 351. Another modification is re-



quired where the access of much or any air would counteract the intention of the operator, by oxidating the body. In such cases the lower end of the tube is either completely closed, or drawn out to a fine orifice as at c in the same figure. A tube of this form is well adapted to the sublimation of selenium from a sulphuret, where the entrance of much air would oxidize it. Decrepitating substances should also be heated in tubes closed at one end, and should be so inclined as to avoid loss of particles. For such and many other purposes tubes, Fig. 168, enlarged into a bulb at one extremity are very appropriate.

All the glass tubes and vessels employed in this way should be perfectly free from lead.

The following instruments are also used in making examinations with the blowpipe. Steel forceps with the points made of platinum, for holding the assay in the blowpipe flame to as-

certain its fusibility or other properties when exposed to an elevated temperature. The two upper figures of the cut represent different views of an excellent forceps, capable of very general application. Two strips of steel, with narrow platinum points b, b, are fastened in the middle by a piece of metal seen at e, e. These strips separated, as seen in the



figure, constitute a double pair, one being at a, a, and the other at c. The platinum points by the elasticity of the metal of which the forceps are made, are always closed. To open them it is only necessary to compress with the thumb and finger, the small projections with the button heads d, d, which are connected with the strips opposite to them. Upon relaxing the pressure, the assay is forcibly held between the points. The points a a are tempered, and are used for detaching exceedingly small fragments of the mineral.

Another form of this instrument is employed, but its use is not quite as convenient as that of the one just mentioned. Its points b c are also of platinum, but curved a little, as represented in the figure. The legs are made of brass. The forceps is kept open by the elasticity of the metal, and closed by a double button d, which slides up and down in a slit cut in the legs. As brass is a good conductor of heat, two pieces of wood e c are fixed to the legs, by which the instrument is held, to prevent any inconvenience to the hand. Under this

last forceps, is still another made, of iron, which can be used for a variety of operations, and which is not solely confined to this application. Substances to be held very firmly are placed between the points. It has a button d d, with a steel spring d e, to prevent the forceps from opening by the sliding back of the button.

A *Microscope*.—A plano-convex microscope, with two lenses of different magnifying powers, is often useful in minute ana-



lysis, and one is represented in Fig. 353, which is made to fit in a small receptacle. By its aid, the minute structure of bodies, and fine colors imparted to the fluxes or to charcoal, which often deceive the naked eye, are examined.

Charcoal Borer.—A conical tube of tinned iron with the margin filed to a sharp edge, for making cavities in the charcoal support, is often made to occupy a place in blowpipe apparatus. It answers very well as a case to contain a phial as shown in the figure above.

A pair of cutting pliers is used to clip off small particles



of minerals, and pieces of a metal or alloy for examination, and for many other purposes which will suggest themselves

to the experimenter. A clasp is attached to the handles for the purpose of keeping them forcibly closed.

The Hammer and the Anvil.—A polished hammer of hardened steel, Fig. 355, with a square, even surface at one end,



and the other terminating in an edge with sharp corners, is a very necessary implement. The flat surface is very applicable for flattening globules of reduced metals, and the edge for breaking off small pieces of minerals. Very small fragments can be broken off without doing any injury to the remaining portion, which is often kept as a specimen.

A necessary accompaniment to the hammer is the anvil,



which is represented in Fig. 356, in a most convenient and compact form. It is made of steel, and is usually about three inches long, one inch in thickness and five-eighths of an inch in breadth, and any one of its surfaces can be used. The substance to be

broken up, or the metallic globule to be flattened out, is enclosed in thin paper, and having been placed upon the anvil, is struck with the hammer until the proper effect is produced. If the substance is reduced to powder, the paper prevents any of it from being scattered or lost.

The Mortar and Pestle.—These implements, made of agate, are of small size, and have been described at page 79. They should be hard and perfectly free from holes and cracks, or they will be liable to fracture and to the filling up of their crevices with the powdered materials—much to the detriment of future operations.

An Electroscope and Magnetic Needle Case.—A cylindrical wooden box is used to contain Haüy's electroscope and a magnetic needle.

The former consists of the hair of a cat, insulated by being inserted in sealing wax poured into the bore of a small glass tube. This tube is fastened in a wooden screw, which closes one end of the case. It is so delicate that a very small quantity of electricity is discovered by its aid. On bring-

ing it near to an excited body, it is attracted by it; but if negative electricity is developed in it, by rubbing or drawing it rapidly through the fingers, and it is then brought in proximity to the excited body, it will be attracted or repelled in accordance with the existence in that body of positive or negative electricity. In the screw at the other end (each one serving as a stand), is fixed a similar tube and sealing wax to insulate a small steel pin, which supports a magnetic needle contained in the box. The needle is mounted with an agate cup to prevent friction as much as possible, when suspended on the point of the pin. In this condition, it is used to indicate the presence of iron when it exists in a mineral in an appreciable quantity, and also the magnetic condition of iron ores. Minerals, before and after being submitted to the action of the blowpipe, should be examined in regard to these properties.

A Steel Magnet.-This is employed in the mode recommended by Hauy to ascertain whether the slightest trace of magnetic force exists in minerals, and consequently whether the metals in which that force exists are present. The experiment is thus performed. The magnet is placed at a small distance from a suspended magnetic needle, its north pole being directed towards that of the needle; it is then gently moved around the needle until the latter takes a position at right angles to its former place, owing to the repulsion of the same kind of magnetism. This repulsion, and the force of terrestrial attraction which tends to make the needle return to its former direction, now hold the needle exactly balanced between them, so that the smallest disturbing magnetic force moves it out of its place. In this way an amount of magnetic influence may be detected, which would not be sufficient to affect the needle in its ordinary state. In performing this experiment, care must be taken not to excite electricity in the mineral by friction, as that force might affect the result more or less.

A knife of good hardened steel is used for trying the com-

Fig. 357.

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parative hardness of metallic bodies and minerals generally. It may be used as a charcoal borer, and if well magnetized can be substituted for the magnet. The point is used to take up the fluxes before mixing them in the palm of the hand with the mineral which has been pulverized for examination.

Files are convenient for detaching small particles of a metal which is to be investigated, cutting glass tubes, and for trying the hardness of bodies. They may be of different shapes, and should be kept clean and out of the reach of corrosive vapors.

An *Edulcorator* or spritz, Fig. 319. This is used to wash the charcoal from the reduced metal. It is necessary to be very cautious in doing this when the metal is small in quantity, as the force of the jet may carry the latter away with the charcoal. A pipette or dropping tube, made by drawing out in the flame of a candle or spirit lamp one end of a glass tube to a small opening, can be used with more impunity. The separation will be facilitated by reducing to powder, in the agate mortar, the charcoal adhering to the piece of metal, as the globule, if malleable, will be thus slightly flattened and made more distinctly visible.

Small capsules of porcelain or watch glasses, are useful for receiving temporarily the results of the experiments; such as specimens of reduced metal, the colored beads, &c., and for keeping separately, different fragments of the minerals to be investigated.

A small pair of scissors, a thin saw with fine teeth for sawing pieces of charcoal, a pair of small tongs for holding crucibles, &c., over the spirit lamp, a small capsule of platinum, a touchstone with needles of gold and alloys of different standards for trying the fineness of gold, will all be found of occasional use.

Fig. 358.

The Box containing the Reagents.—As it is necessary to have the fluxes always ready for use, Gahn contrived a con-

#### BLOWPIPE MANIPULATION: THE REAGENTS.

venient and portable box for the purpose, which is seen in Fig. 358. It is  $8\frac{1}{2}$  inches long,  $1\frac{3}{16}$  broad, 1 inch in height, and is divided into nine compartments to receive the different reagents. Each division has a lid nicely closing its particular box so as to prevent any one substance from becoming mixed with the others. A common lid closes over these smaller ones and is fastened to the box by two hooks. The cross pieces, which are permanently fixed to the large lid, fit into spaces between the 2d and 3d lids from each end, and serve to make them more secure. If more reagents are required than can be contained in these boxes, those which are but seldom used may be wrapped in paper and placed in one of them.

The Reagents.—The reagents, which must all be chemically pure, are the following:—

Carbonate of Soda, commonly called soda, which is much used to detect the presence of silica, to assist the reduction of metallic oxides, and generally, to determine whether a body unites with it to the production of a fusible compound.

Cyanide of Potassium.—This substance being very deliquescent, should be kept as free as possible from contact with humid air, and had better be placed in a small, tightly corked test tube, which may have its place in one of the small compartments of the box.

As a blowpipe reagent, cyanide of potassium is highly useful; its action is indeed extraordinary. Substances like peroxide of tin, sulphuret of tin, &c. &c., which for their reduction with carbonate of soda, require rather a strong flame, are reduced with the greatest facility when cyanide of potassium is used. In blowpipe experiments we always use a mixture of equal parts of carbonate of soda and of cyanide of potassium, since the latter alone fuses too easily. This mixture, besides its more powerful action, has another advantage over carbonate of soda: it is with extreme facility imbibed by the porous charcoals, so that the purest metallic globules are obtained.

Biborate of Soda.—This salt, which is commonly called borax, is used to facilitate the fusion of very many substances. When melted with the metallic oxides, its bead assumes a great variety of colors, which furnish excellent indications of the presence of the metals.

Phosphate of Soda and Ammonia.—This substance, called also microcosmic salt, phosphorus salt, and fusible flux, is of very general application, and as it dissolves most of the me-

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### BLOWPIPE MANIPULATION: THE REAGENTS.

tallic oxides with great readiness, the colors produced in its bead are, if possible, more brilliant and characteristic than those made with borax.

Nitrate of Potassa, or saltpetre, is used to assist in the oxidation of metals, as it yields up its oxygen very readily when exposed to heat.

Bisulphate of Potassa in solution, is used to indicate lithia, boracic acid, nitric acid, fluohydric acid, bromine and iodine; and also for the separation of baryta and strontia from other earths and metallic oxides.

Vitrified Boracic Acid (glass of borax) is used to detect the presence of phosphoric acid; also small portions of copper in alloys of lead.

Fluoride of Calcium (fluor spar), when mixed with bisulphate of potassa, serves to detect lithia and boracic acid. Alone it is a test for gypsum.

Sulphate of Lime, or gypsum, in the form of plaster of Paris, is sometimes used as a reagent with fluoride of calcium.

Nitrate of Cobalt.—This very valuable test is used in a somewhat concentrated solution.

Alumina heated in the oxidating flames, after being moistened by a drop or two of this solution, acquires a beautiful pale blue color; magnesia a rose red tint, and zinc a bright

green. The solution is contained in a phial similar Fig. 359. to the one represented in Fig. 359. The glass stop-



ple, tapering to a point, descends into the solution, so that on withdrawing it, a small quantity adheres to its extremity. Berzelius uses a cork stopple with a platinum wire flattened out in the form of a spoon, at the end which is immersed in the solution. The phial may be of such a size as to be conveniently received in the charcoal borer, page 381. Oxalate of cobalt may be made to take its place, and as it is

used in powder, it is often of more convenient application.

Nitrate of Nickel in solution, or Oxalate of Nickel in powder. The oxide of nickel gives a brown color to soda glass, while potash, if melted with a substance containing it, acquires a bluish purple color. A bottle similar to the one just described may contain the solution of the nitrate.

*Lead*, very pure, and especially free from silver, is used in cupellation.

Bone ashes are employed in cupelling metals containing gold and silver, or some of the ores. The cupels are prepared

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#### BLOWPIPE MANIPULATION: THE REAGENTS.

by moistening a small quantity of the ashes, mixed with a little soda salt to make it coherent, and by kneading the mass in the palm of the left hand to the consistence of a stiff paste. A cylindrical hole made in a piece of charcoal is then filled with the paste, and after the surface is smoothed with the small agate pestle, a slight depression is made in the centre, sufficiently large to hold the metal or mineral to be cupelled, together with a small quantity of the proof lead. The cupel is slowly dried by heating it carefully in a stove or over the flame of a spirit lamp. The assay with the lead is then placed on the cupel and submitted to the action of the exterior or oxidating blowpipe flame. By the influence of this, the lead is oxidized, and the fused litharge so formed, is absorbed by the bone ashes, while the silver or gold is left behind in the form of a brilliant globule; which, before its complete purification, exhibits the iridescence formerly described under CUPELLATION. Plattner describes a convenient instrument for making the cupels.

Oxide of Copper is used for the purpose of detecting chlorine.

Silicic Acid, when melted into a fusible glass with soda, is a test for sulphur or sulphuric acid. The assay must, however, not contain it.

*Silver*, in the form of wire or foil, is made use of for ascertaining the presence of sulphuret of potassium, or any other soluble sulphuret.

Tin Foil sometimes assists in the reduction of metallic oxides, which are dissolved in a bead of one of the fluxes, and by its use we sometimes get a more satisfactory result than is obtained without it. For instance, when a small quantity of copper is dissolved in a bead of borax, or of microcosmic salt, and the glass is treated in the reducing flame, it sometimes becomes ruby red and opaque. But if the amount of copper is so small that the reducing flame cannot produce this result, a little tin added to the bead, and heated with it, makes the proper appearance evident immediately upon its cooling.

Iron wire, which is generally that metal in its purest state, precipitates some other metals from the different fluxes, or separates therefrom, sulphur and the fixed acids. It is also used to reduce phosphoric acid to phosphorus, which combining with iron, forms a white brittle metallic globule, the phosphuret of iron.

Besides the above mentioned tests, it is proper to have

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Formate of Soda, which, when anhydrous, is used to detect arsenic in oxide of antimony. Test papers colored with litmus, Brazil wood, and turmeric, are also convenient.

The substances mentioned in the foregoing list as reagents are all of those which are essential to the completeness of the blowpipe apparatus. While, however, occasions may arise for the use of any or all of them, the great majority of examinations with the blowpipe can be made with the aid of but a few, and the possession of the first four or five upon our list, with the fluid nitrate of cobalt and the metals referred to, may be considered as quite enough to make the manipulator competent to pursue ordinary investigations.

Blowpipe Table.—In the Laboratory all the instruments essential to the expedition of blowpipe analysis are placed within convenient reach of the operator. For this purpose Gahn's table, which has drawers both in the side and front, will be found very useful. The side drawers are divided into many compartments, and are shown in Fig. 360, drawn out from their usual position. The right hand drawer contains



the apparatus most frequently used, and the left that which is less often required. The lamp, blowpipe, fuel, wick and other necessaries of a rougher kind occupy those in front.

This table, although quite small, may be found to take up too much room. Berzelius' case, which is much more portable, may then be substituted for it. It consists of a neat

#### BLOWPIPE.-THE TRAVELING CASE.

mahogany case, exhibited in Fig. 361, which has a cover, and is 14 inches long and  $9\frac{1}{2}$  inches wide. Each article is made to fit closely in a corresponding cavity, so that it is kept firmly in



its place. It contains all the necessary apparatus for these experiments, and scarcely occupies more space than an ordinary portfolio. It is neatly put up by Kent, in New York, with the apparatus and tests, after directions given by Berzelius.

Traveling Case.—Although Berzelius' case occupies little space, and can readily be introduced into a common trunk, many mineralogists prefer having their blowpipe apparatus enclosed in a still more compact form. The traveling case is arranged very much in the same way as the large size of surgical instrument cases, and consists of a piece of leather forty inches or more in length, with sides capable of being folded down upon the body, and long strips of the same material tacked along its centre, so as to leave open spaces for the insertion of the various pieces of apparatus. After these latter have been deposited in place, the lateral folds are closed upon them, and the whole is rolled up and tied with tape, or brought together with a common strap and buckle.

It is advisable to place the largest instruments near that end of the case which is first rolled up. When blowpipe operations are in progress, the case can be unrolled and suspended from a nail in a wall, so that free access can be had to all its contents.

Besides the various parts of apparatus which we have been describing, it is well that the operator should be provided with a piece of sheet iron twelve inches or more in diameter, and with a rim turned up around the margin. This may be placed upon the table, under the lamp, and will serve to retain ignited or other particles thrown off from the substance which is under examination. A sheet of white paper placed over it will enable the experimenter to discover with ease, minute particles which might otherwise be lost.

It will be well for the chemist to supply himself with a set of chemically pure tests which may be kept in small stoppered vials. Such a series is very useful as affording the means of comparing the behavior of a mineral with that of the substance supposed to be an ingredient of it, and of thus verifying results. Its possession, moreover, conduces to the attainment of dexterity in manipulation, and of the knowledge of the various reactions occurring under the blowpipe flame.

The set may consist of-

Alkalies Baryta Strontian Lime Magnesia Alumina Glucina Yttria Zirconia Thorina Silica Acids of Arsenic Vanadic acid Molybdic acid Tungstic acid Oxide of chrome Antimony and its oxides Oxide of tellurium and telluric acid Tantalic acid Titanic acid Oxides of uranium Oxides of cerium Oxide of lantanium Oxide of didymium Oxide of manganese Oxide of zinc Oxide of cadmium

Oxide of iron Oxide of cobalt Oxide of nickel Bismuth and its oxide Oxides of tin Oxide of lead Oxide of copper Mercury Oxide of silver Sulphurets Seleniurets Arseniurets Antimoniurets Tellurets Carburets Sulphuric acid Nitric acid Chlorides Bromides Iodides Fluorides Phosphates Carbonates Boracic acid Hydrates Silicates Salts of metallic acids

It has only come within our province to describe the implements and adjuncts employed in blowpipe analyses, with some few examples of the practical results obtained by means of them. The excellent manual of Griffin, and the works upon the subject by Berzelius and Plattner, contain most complete accounts of this branch of manipulative chemistry, and of the mode of conducting investigations by means of it.

# CHAPTER XXVIII.

# APPLICATION OF THE CIRCULAR POLARIZATION OF LIGHT TO THE ANALYSIS OF SACCHARINE SUBSTANCES.

WHEN a ray of common light passes through a doubly refracting crystal, such as a rhomb of Iceland spar, it is separated into two rays which have peculiar properties differing from the original ray. These rays are found to possess similar properties in planes at right angles to each other; that is if we suppose one ray to have certain properties in a horizontal plane, the other ray will have similar in a vertical plane. Each of these rays is said to be polarized, and to have its plane of polarization at a right angle to that of the other.

If one of these rays be absorbed or prevented from passing by any means, and the other whose *plane of polarization* we will suppose to be horizontal, be allowed to pass through another doubly refracting crystal, as Iceland spar, in certain positions the polarized ray will be again doubly refracted, and we shall see two images of the object from which the light comes. But if we turn the second crystal in such a position that its *principal section*, that is the plane passing

through the axis A  $\hat{X}$ , (which is the line joining the obtuse summits of the rhomb,) and perpendicular to one of its faces, is vertical, that is at right angles to the plane of polarization of the ray, only one ray will pass through the crystal, the other being stopped, and but one image will be seen. If now we continue to turn the second crystal, the remaining ray decreases in intensity, and the other begins to reappear, and if we go on turning the



crystal through 90°, the first ray will disappear and the ray which had formerly disappeared will be at its maximum intensity. If we turn the crystal through another 90°, this ray will again disappear and the other reappear. And so on these changes alternate through every 90°, until finally we come again to our original position, with the principal section at right angles to the plane of polarization.

Now it is evident that if we should stop one of the two rays into which the second crystal refracts the polarized ray, then in certain positions we should have no light transmitted, and in positions at right angles to these, we should have the greatest intensity.

The first of these crystals of Iceland spar is called the polarizing crystal, or *polarizer*, and the second the *analyzer*. When we make use of two *Nichol's prisms*, (which are rhombs of Iceland spar so contrived as to transmit only one of the doubly refracted rays,) one as the polarizer, and the other as the analyzer, and the analyzer is turned until its principal section is at right angles to the plane of polarization of the polarized ray, no light passes. This position is called the *azimuth zero*. When the analyzer is turned through 90° from this position the polarized ray attains its greatest brightness. Turning again 90°, the light disappears, and reappears on turning through another 90°, and finally again disappears in returning to the azimuth zero.

If when the polarizer and analyzer are in this position, a piece of quartz which has been cut from a crystal at right

Fig. 363.



angles to its axis A X, or a solution of cane sugar be placed intervening, so that the polarized light has to pass through them, the light immediately reappears, with a tone of color depending upon the thickness of the section of quartz, or solution of cane sugar. If we turn the analyzer in a direction from *left* to *right*, then if the original color was orange, for example, we shall find the prismatic colors succeeding each other in the order of

orange, yellow, green, blue, indigo, violet, red. When the colors succeed each other in this order, the quartz and the sugar are said to deviate or rotate the plane of polarization to the *right*. When by turning the analyzer in the same direction the colors succeed each other in *inverse order*, or when by turning the analyzer from *right* to *left* the colors succeed each other in the same order, the quartz and the sugar are said to deviate the plane of polarization to the *left*. In the former case the quartz and sugar are said to be *right*- handed, in the latter *left-handed*. Crystallizable sugar deviates the plane of polarization always to the right, or is right-handed, uncrystallizable to the left or is left-handed. Some specimens of quartz are found to deviate the plane of polarization in one direction, and some in the opposite.

When the analyzer is not a Nichol's prism, but only an ordinary doubly refracting prism, which allows both rays to pass, and is in the position in which one of the rays into which the polarized beam is refracted is stopped, then the

plate of quartz will cause it to reappear, and we shall have the two beams o and e, Fig. 364, colored but not with the same tint, one complementary to the other. Two colors are said to be complementary if they produce white light when mixed.

These two colors vary as we rotate the analyzer, but in all cases they are complementary.

If when the analyzer is at the azimuth zero, we substitute for the simple plate of quartz, a plate composed of *right* and *left*-

handed quartz, R and L, Fig. 365, the line of separation of which is vertical, then as they are of the same thickness, we shall have the same appearance and colors in the two rays, as we had in the case of the simple plate of quartz of the

Fig. 366.

same thickness. For the two kinds of quartz being of the same thickness, the one deviates the plane of polarization as much to the right as the other to the left. Hence each one of the rays, although of complementary colors, will be of the same uniform color in itself.

But if the analyzer be turned each beam will be divided into two different colors, l, r, and l, r', Fig. 366, and the colors in one beam will be complementary to the colors in the other beam.

If two plates of quartz of equal thickness, one right-handed,





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Fig. 367.

### VENTZKE'S APPARATUS.

the other left-handed, be made to overlap, Fig. 367, then the effects of each separately, will be neutralized, and the plane of polarization will not be deviated in either direction; and if the analyzer be at the azimuth zero, only one ray will pass, as if the quartz had not been interposed.

Having now prefaced with the phenomena of polarized light employed in chemical analysis, we shall describe the apparatus of M. Ventzke, which is a modification of the original apparatus of M. Biot, who first discovered the property of the circular polarization of light possessed by sugars, and other organic matters, and who founded on this discovery his process of analyzing solutions of saccharine substances. This apparatus was used by Prof. McCulloch in quite an extended series of analyses for the government of the United States.\*

A sketch of the apparatus is given in the annexed cut. The



analyzer, which is a Nichol's prism, is placed in a brass socket at A, another view of which is given at A (Fig. 369). This socket is capable of a motion in the graduated disk and carries an index I, which moves over the disk and measures the number of degrees from the zero point, through which the analyzer is moved. It is turned by means of a small disk F, called the pinion disk, which carries a pinion wheel gearing into a larger toothed wheel attached to the analyzer.

The polarizer, which is also a Nichol's prism, is fixed in a brass socket at B. This socket has a toothed wheel which

\* Senate Document 165, 28th Congress, 2d Session.

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gears into the threads of a perpetual screw, which is moved by a milled head at b. The whole socket is capable of motion in a groove, and of being fixed in any position by the binding screw d. The tube which is to contain the solution of sugar is shown at D. The peculiar construction of this tube will be explained directly. A lamp is placed at E, which is recommended to be used instead of the sun, in order to insure a uniform intensity of light. The whole instrument is mounted on an iron foot.

The only adjustment necessary is to bring the principal section of the analyzing prism at right angles to the plane of polarization of the polarizer. This is done by putting the index of the analyzer at zero, on the graduated arc, and then, by means of the screw b, turning the polarizer until we have obtained the point of greatest darkness. This adjustment is best made by sun light. This gives the azimuth zero.

It has already been mentioned, that crystallizable sugar deviates the plane of polarization to the right, and uncrystallizable to the left. Now, as the sugars, which in the majority of cases the chemist is required to analyze, contain the two kinds, it becomes necessary to appreciate the

effect of each kind separately in deviating the plane of polarization. It has been found that when hydrochloric or sulphuric acid is added to a solution of pure cane sugar, which always polarizes to the right, and the mixture is suffered to stand for 10 or 12 hours, or is gently heated, the cane sugar is converted into the uncrystallizable, which deviates to the left. or is left handed. Advantage is taken of this fact in M. Biot's process of analysis. A solution is made which contains 25 or 50 per cent. of the sugar to be analyzed. This solution is then filtered, and if it is much colored it is decolorized by passing it through a tolerably coarse bone black. It is then placed in a glass tube a, Fig. 370, the ends of which are ground, and are fitted with screws as shown in the figure. Over the end of the tube is placed a round disk of



glass, b, with parallel surfaces, and then the cap c is screwed down over this so as to hold it tightly in its place. A hole is

pierced through the cap at d, which allows the light to pass. The other end of the tube is provided with a similar arrangement. This tube having been filled with the solution is placed in the apparatus in the position shown at D. The analyzer is then turned to the right until the bluish violet ray begins to appear. This color is chosen because it is one of the most delicate, and its amplitude is small, and therefore is observed with the greatest certainty. This angle is noted. Then one part by volume of hydrochloric acid is added to nine parts by volume of the solution, so that the original sugar solution becomes  $\frac{9}{10}$  of the acidulated solution. This is then suffered to stand for 10 or 12 hours, or if gently heated to 154° Fahr., is entirely converted into uncrystallizable sugar in 15 or 20 minutes. It is again placed in a tube, and the analyzer turned to the left until the same bluish violet tint appears. This angle is then noted. Previously to adding the acid, the density of the solution is observed either by a good hydrometer, or by weighing.

Calling the angle observed before acidulation the *direct* angle, the angle observed after acidulation the *inverted angle*, and the ratio of the original solution to the acidulated mixture, which in the above example is  $\frac{9}{10}$ , the *ratio of dilution*; then for convenience we may take the notation.

$$a = \text{direct angle},$$

- a'' =inverted angle,
- n = ratio of dilution = 0.9,
- d = density of the solution,
- e = per cent. of the original sugar contained in the solution, which is either 25 or 50,
- z = the per cent. of pure cane sugar in the original sugar.

Knowing the first five quantities, we may calculate the last by the following rule:\*

\* The formula which Mr. M'Culloch has given at p. 24 of his Second Report, Senate Doc., No. 209, 29th Congress, 2d Session, is

$$=\frac{s}{153.76 \ n \ e \ d}$$

in which

$$s = \frac{1 + r''}{1.38} a'$$
, in which  $a' = n a$ , and  $r'' = \frac{a''}{a'}$ 

the same notation as given above.

Multiply a by n, with the product thus obtained, as a divisor, divide a'': add 1 to the quotient, and multiply by the product of a and n; divide the product thus obtained by 1.38: divide the quotient by 153.76 multiplied by n, by e and by x; the quotient will give the value of z, or the per cent. of pure crystallizable cane sugar, contained in the original sugar.

In analyzing molasses the same process is followed. The only difficulty which occurs is the decolorization of the solution; which may be done by repeatedly filtering through tubes filled with coarse bone black. A glass tube of  $\frac{1}{2}$  or  $\frac{3}{4}$  inch interior diameter, and about 2 or 3 feet long, is taken, which is narrowed at one end by the lamp and blowpipe; a loose plug of paper is then put in the tube near this end, and the tube is filled with coarse bone black. It is recommended by M. Clerget not to collect the first part of the filtrate, as the bone black absorbs some sugar as well as coloring matter, and thus alters the per cent. of the solution. He recommends that a volume of the solution about equal to that of the bone black should be lost. The liquor, if not sufficiently decolorized by the first filtration, may be passed through the same black again.

Other processes may be employed which are known to chemists, such as precipitating the extractive and coloring matters by the subacetate of lead. In this case the solution of the subacetate should be used as pure water, in making the original solution. In defecating cane juice M. Clerget proceeds as follows: He prepares a solution of isinglass (fishglue) by macerating 5 or 6 grammes of it in 250 grammes of cold water during 3 hours. He thus obtains a paste, which is sifted or strained through a piece of coarse linen, and mixed with 100 grammes of white wine or alcohol diluted with water. Thus is obtained a gelatinous mass which may be diluted with water until its volume is 1 litre (61 cubic inches). This will keep in a corked bottle from 15 to 20 days, according to the temperature. It should not be used when it becomes very sour. If a small quantity of this be added to cane juice, or any solution of sugar and mixed with it and then the same volume of alcohol be added, the whole is coagulated, and the solution is left perfectly clarified. The dilution of the solution which thus takes place must be taken into account in the calculation of the per cent. of sugar dissolved. It may be

compensated by increasing the length of the tube in the same ratio.

In the October number of the Bulletin de la Société d'encouragement pour l'Industrie Nationale, 1846, M. Soleil, the celebrated optician of Paris, has described a nouveau saccharimètre of his invention, which is a great improvement over the instrument which has been described, enabling the measurements to be made with great precision. The instrument is represented in the cut, Fig. 371.

The polarizer is placed at d, which is either a Nichol's prism, or an ordinary doubly refracting prism. The analyzer is at a, which is a doubly refracting prism, which separates the polarized beam into two, and in certain positions, allows only one to pass. Immediately behind the polarizer at e, two pieces of quartz of equal thicknesses, one right-handed. the other left-handed, are placed, so that the line of separation is vertical, and in the middle of the field of view, so that one acts on one-half the beam, the other on the other half. Now, the analyzer being at the azimuth zero, from what has been said, the same tone of color will be produced by each, because they rotate the plane of polarization equally and in opposite directions. Hence, although the two beams into which the analyzer will separate the polarized light will be of complementary colors, yet the color of each beam will be uniform throughout. Now when the sugar solution is placed at n, if it is crystallizable sugar it will be like increasing the thickness of the right-handed, and diminishing that of the left-handed quartz; if uncrystallizable, like increasing the thickness of the left-handed and diminishing that of the righthanded quartz. Hence, in either case the plane of polarization will be rotated more in one way than in the opposite, and the amount of this rotation will depend upon the number of particles of sugar, that is upon the per cent. of the solution, and the length of the tube; and, therefore, each of the two beams will be no longer of the same uniform tint, but one-half of one beam will be of one color and the other half of the other color, and the other beam will also have two colors complementary to those of the first.

In order to measure the degree to which the plane of polarization is rotated in either direction, M. Soleil ascertains the thickness of the quartz of either kind, which is necessary to compensate the effect of the sugar that is to render each beam

# SOLEIL'S SACCHARIMETER.



### SOLEIL'S SACCHARIMETER.

of a uniform tone of color. This is accomplished by his *compensator*, which part of the apparatus is placed at c.

Now the effect of the sugar in deviating the plane of polarization depends upon its purity and upon the strength of the solution; therefore, in order to compensate the effect of the sugar in every case, it would be necessary to have a number of pieces of quartz of various thicknesses, and of contrary effect to the sugar, in deviating the plane of polarization. This would be inconvenient, and M. Soleil overcomes



the difficulty in his compensator. Immediately before the sugar solution, and near c, is placed a piece of quartz, which rotates the plane of polarization to the right. At c there are two pieces of left-handed quartz, cut as shown in Fig. 372. Now



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## SOLEIL'S SACCHARIMETER.

when these two pieces a and b are in the position shown in the figure, they have together the same thickness as R, and being of an opposite kind to R, of course they neutralize its effect. But when they are in the position shown at c d (Fig. 373), then their thickness in the direction of the ray l l, is less than the thickness of R, and, therefore, the effect of R will predominate, and it will compensate for a left-handed (uncrystallizable) sugar. When, however, the pieces are in the position shown at e and f, then the thickness through l l is greater than that of R, and their deviation will predominate, and will compensate for a right-handed (crystallizable) sugar.

These two pieces are mounted upon a small rack work into which gears a pinion, which moves them in opposite directions. The pinion is turned by a milled head at b. These two pieces have likewise attached to them two scales, which are represented at c c'. The smaller scale serves as a vernier, 10 of whose divisions correspond to 9 on the larger scale. They are each graduated in opposite directions from a common zero point, and the extremities of both scale and vernier are marked D, *droit*, *right*, and G, *gauche*, *left*. When the zero points coincide, the two pieces are in the position shown at a and b, and have the same thickness taken together, as R.

Each division of the large scale counts 10, and each of the small scale 1. Thus if the zero point of the vernier was between 4 and 5 on the large scale, very near 5, we glance along the vernier, until we find a division which coincides with a division on the large scale. If this division is 9, then the instrument gives the reading 40 on this large scale, and 9 on the vernier, or 49.

A lens is placed at g, which is to be so adjusted as to see clearly the vertical division line of the two quartz placed at e. This adjustment should be made while the tube u is full of water. A spiral spring is placed at q to press against the tube u, and keep it in its place. The whole instrument is mounted on a pillar and foot.

Now supposing the lens at g adjusted so as clearly to see the line of separation of the two pieces of quartz at e, and the scale and vernier adjusted to their common zero point, the only remaining adjustment is that of the analyzer to the azimuth zero, which may be done by rotating it in its socket until one of the images of the aperture at o has a uniform violet color. When this is done, the analyzer is fixed firmly in its place by means of the binding screw p. In this apparatus a tube of the solution is used as in the former case, and in order to determine the amount to which it deviates the plane of polarization, the button at b is turned in whatever direction tends to produce a uniform violet color in the colored ray which was uniformly violet before the solution was placed in the apparatus. Then the direction in which the vernier has moved will determine the kind of deviation: if towards D the plane of polarization is rotated towards the right; if towards G, to the left.

Analyses may be made in precisely the same way, and calculated by the same formula with the exception of the numerical factor 153.76, which will have to be determined anew, as it depends on the instrument.

M. Clerget has given a table which supersedes the necessity of calculation.\* His process of analysis is as follows :---Having made a solution containing 16.471 per cent. of pure dry cane sugar, and placed it in a tube 20 centimetres (7.8 inches) long, he found that it deviated the plane of polarization to the right 100°. Now if we were analyzing a sugar known not to contain any left polarizing substance, and we should find that it deviated the plane of polarization 80° to the right, then by stating the proportion :

# 100: 16.471:: 80: x = 13.177.

Now dividing 13.177 by 16.471, we get the per cent. of pure cane sugar in the original sugar. If, instead of making this calculation, we refer to the table at the end of this article, and look down the column A to the number 80, the number in column B on the same horizontal line, is the number of grammes and centigrammes contained in a litre of the solution. The solution is supposed to be observed through a tube of a constant length, 20 centimetres.

As the sugars to be analyzed generally contain left polarizing sugar, it is necessary to take into consideration the effect of this substance. A solution of the sugar or molasses is made of the normal per cent. 16.471, and decolorized and clarified according to the methods described. A tube 20 centimetres long of the solution is then placed in the instrument and the direct deviation noted. Ten volumes of the solution are then added to one volume of concentrated hydrochloric acid,

\* Bulletin de la Société d'encouragement pour l'Industrie Nationale, Oct. 1846.

## CLERGET'S METHOD.

and then put into a convenient vessel (a matras is best adapted to this purpose), and the whole placed in a water bath and brought up to the temperature 68° cent. (154° Fahr.). The heat is so regulated as to require about fifteen minutes to bring it to this temperature. It is then placed in a vessel of cold water, in order to bring it to the temperature of the surrounding air, and afterwards in a tube, represented in Fig. 374, adapted with a thermometer, so as to take the tempe-



rature of the liquor at the time the inverted angle is observed in the polarizing instrument; M. Clerget having found that the temperature, at which the observation is made, has a great influence on the deviation of the plane of polarization. Having increased this last number by  $\frac{1}{10}$ th in order to compensate for the dilution by the acid, it is added to the direct deviation, and then entering the table, at the temperature at which the inverted deviation was noted, we find the number nearest to the sum, and the number in the column A on the same horizontal line, will give the per cent. of pure sugar.

In the case, where the deviation before the acidulation and after, takes place in the same direction, which may happen if the crystallizable sugar is mixed with a large quantity of uncrystallizable, then the difference instead of the sum of the deviations is to be taken.

*Examples.*—Suppose a liquor before acidulation gives a direct deviation of  $75^{\circ}$ and after acidulation, an inverted deviation at the temperature of  $15^{\circ}$  cent.  $20^{\circ}$ 

Sum

95°

# CLERGET'S METHOD.

Entering the table at the column of 15° centigrade, we find 95.5 corresponds to 70 per cent. of cane sugar.

Again, suppose before acidulation the direct deviation is and after at 20° cent. the *direct* deviation is  $-26^{\circ}$ 

Difference - - - - 54° In the column 20° cent., 53.6 gives 40 per cent. cane sugar.

Those who wish to understand fully the application of the phenomena of circular polarization to chemical analysis, are referred to the memoirs of M. Biot in xiii. xiv. xv. vols. of the *Mémoires de l'Académie*; to Professor McCulloch's Report, Senate Documents, No. 165, 28th Congress, second session, or the Scientific Memoirs, vol. iv. p. 292, which contains a translation of some of M. Biot's papers.

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TABLE FOR THE ANALYSES OF SACCHARINE SUBSTANCES.

ANALYTICAL TABLES.

405

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ANALYTICAL TABLES.

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Per	by wt.	82886669282828282666664444444448666668888888888
T.)	35°	41.7 441.7 441.7 441.7 445.5 55.7 55.7 55.7 55.7 55.7 55.7 5
(CEN	340	41.9 441.9 441.9 445.744.7 445.7 445.7 445.7 445.7 445.744.7 445.7 445.7 445.7 445.744.7 445.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.744.7 445.7 445.744.7 445.7 445.744.7 445.7447
RES	330	42.1 44.6 44.6 44.6 44.6 44.6 44.6 44.6 44
ATU	320	449.5 449.5 449.5 449.6 449.7 449.7 449.7 449.7 449.7 449.7 55.6 55.7 55.6 55.7 55.6 55.7 55.7 55
IPER	310	42.44 44.45 45.0 44.45 45.0 44.45 55.0 44.75 55.0 55.0 55.0 55.0 55.0 55.0 55.0 5
TEN	300	42.6 43.6 44.5 44.5 44.5 44.5 44.5 44.5 44.5 44
THE	290	42.0 42.0 42.0 44.0 44.0 44.0 44.0 44.0
AT	280	42.9 44.2 44.2 44.2 44.2 44.2 44.2 44.2
KEN	270	43.1 44.4 44.4 44.4 44.4 44.4 44.4 44.4
I, TA	260	$\begin{array}{c} 43.5\\ 6.5.1\\ 7.5.5\\ 7.5$
ERSE	250	43.4 44.74 44.74 44.74 44.74 44.74 44.74 44.74 44.74 44.74 44.74 66.74 77 66.74 77 66.74 77 66.74 77 66.74 77 66.74 77 66.74 77 66.74 77 76 86.75 86.7
INVI	240	443.0 443.0 444.0 444.0 55.4 444.0 55.4 444.0 55.4 444.0 55.4 444.0 55.4 444.0 55.4 13.5 55.4 13.5 55.4 13.5 55.4 13.5 55.4 13.5 55.4 13.5 55.4 13.5 55.4 14.5 55.4 15.5 55.5 5
AND	230	$\begin{array}{c} 43.4\\ 44.5\\ 45.4\\ 44.5\\$
TOE	22 <sup>c</sup>	43.0 44.0 44.0 44.0 55.0 55.0 55.0 55.0 55
DIRI	210	444.0 454.0 454.4 454.0 454.4 454.0 454.7 550.7 551.7 551.7 752.7 552.7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7
NS,	200	445 45 45 45 45 45 45 45 45 45 45 45 45
ATIC	19c	444 454 454 457 457 457 457 457 457 457
DEV	180	444 444 496 496 665 655 755 655 755 665 77 72 72 72 75 665 75 75 75 75 75 75 75 75 75 75 75 75 75
THE	170	44.47 45.47 46.47 55.555
OF 7	16°	44 44 45 45 45 45 45 45 45 45 45 45 45 4
CES	150	45.40 45.40 45.50 55.100
REN	140	45,444 45,25 55,54755,547 55,547 55,54755,547 55,547 55,54755,547 55,547 55,54755,547 55,547 55,54755,547 55,547 55,54755,547 55,547 55,54755,547 55,54755,547 55,547 55,54755,555 55,54755,557 55,547 55,54755,557 55,547 55,54755,557 55,54755,557 55,547 55,54755,557 55,547 55,54755,557 55,547 55,54755,557 55,547 55,54755,557 55,547 55,54755,557 55,547 55,54755,557 55,547 55,54755,557 55,547 55,54755,557 55,547 55,54755,557 55,547 55,54755,557 55,54755,557 55,547 55,54755,557 55,54755,557 55,54755,557 55,54755,557 55,54755,557 55,55755,557 55,55755,557 55,55755,557 55,55755,557 55,55755,557 55,55755,557 55,55755,557 55,55755,557 55,55755,557 55,55755,557 55,5575755,557 55,55757557 55,55757557557 55,55757575755
IFFF	130	45.4 45.4 45.5 45.5 45.5 45.5 45.5 45.5
I QN	120	445 444 444 444 444 444 444 444 444 444
IS AI	110	4444 4444 85556 85557 8557 8557 8557 8557 8557 85
SUA	100	90.00 00 00 00 00 00 00 00 00 00 00 00 00

cent.	by vol. B	gram. 1105.70 1105.70 1105.70 1105.70 1113.64 1113.64 1113.64 1113.64 1113.64 1113.64 1113.64 1113.64 1113.64 1113.64 1145.95 1135.75
Per	by wt.	853833339398888888888888888888888888888
('LN	35°	83.5 84.5 84.5 88.5 88.5 88.5 88.5 88.5 88
(CEI	340	883.8883.885.4885.4885.4885.4885.4885.48
RES	330	84.1 85.4 85.4 85.7 85.7 85.7 85.7 85.7 99.5 99.5 99.8 99.8 99.8 99.8 99.8 99.8
ATU	320	84.5 84.5 85.5 85.5 86.0 90.6 90.6 99.5 99.5 99.5 99.5 99.5 99.5 99.5 99
IPER	310	84.8 84.8 87.4 87.4 87.4 87.4 91.2 91.2 95.1 95.1 95.1 95.1 95.1 95.1 95.1 95.1
TEN	30°	85.1 56.4 56.4 56.5 56.5 99.6 99.5 99.5 99.5 99.5 99.5
THE	290	86.5 86.5 88.1 88.1 90.6 91.9 91.9 97.1 99.1 99.1 99.1 99.1 10.0 99.1 10.0 10.0
AT	280	85.8 88.4 88.4 88.4 88.4 99.7 99.2 99.8 99.8 99.8 99.8 99.8 99.8 99.8
KEN	270	86.1 87.4 88.7 88.7 88.7 88.7 99.0 99.2 99.2 99.2 99.2 99.2 99.2 99.2
E, TA	260	<ul> <li>\$65.5</li> <li>\$65.5</li> <li>\$65.5</li> <li>\$65.5</li> <li>\$65.5</li> <li>\$65.5</li> <li>\$65.5</li> <li>\$65.5</li> <li>\$90.4</li> <li>\$95.6</li> <li></li></ul>
ERSI	250	861,8 881,1 881,1 881,1 990,7 990,7 990,9 990,9 990,9 990,9 990,9 100,1 111,5 11,5
INV	240	<ul> <li>87.1</li> <li>88.9</li> <li>89.8</li> <li>89.8</li> <li>89.8</li> <li>89.8</li> <li>99.1</li> <li>99.1</li> <li>99.3</li> <li>99.3</li> <li>97.7</li> <li>99.4</li> <li>99.6</li> <li>99.6</li> <li>99.6</li> <li>99.6</li> <li>99.6</li> <li>99.7</li> <li>99.7</li> <li>99.7</li> <li>99.8</li> <li>99.8</li> <li>99.8</li> <li>99.9</li> <li>99.9</li></ul>
AND	230	67.4 885.4 90.1 90.1 99.4 99.4 99.4 99.4 99.4 111.3 11.3 11
ECT	220	57,8 89,1,6 99,4,4 99,4,4 97,1,1 99,4,4 99,4,4 99,4,4 10,0,1 10,0,1 10,0,1 11,0,4 11,0
DIR	210	88.1 89.1 99.4 99.4 99.4 99.4 99.4 10.5 99.4 10.5 99.4 10.5 99.4 10.5 99.4 10.5 10.5 10.5 10.5 10.5 10.5 10.5 10.5
ONS,	200	888 4 893 5 991 1 991 1 995 5 995 5 995 5 995 5 995 5 995 5 995 5 100 5 1000 5 100 5 1000 5 100 5 1000 5 10000000000
IATI	19°	83.5 99.5 99.5 99.5 99.5 99.5 99.5 99.5 9
DEV	180	891.5 891.5 995.5 995.5 995.5 995.5 995.5 995.5 995.5 995.5 10000000000
THE	170	80.4 90.5 90.4 90.6 90.4 95.9 95.9 95.9 95.9 95.9 95.9 95.9 95
OF	16°	89.8 91.1 92.5 92.5 93.2 99.3 99.3 99.3 99.3 99.3 99.3 91.0 10.5 1111.5 1111.5 1111.5 1111.5 1111.5 1111.5 1111.5 12.1 1111.5 12.1 1111.5 12.1 1111.5 12.1 1111.5 12.1 12.5 12.5
CES	150	90.1 90.2 90.5 90.5 90.5 90.5 90.5 90.5 90.5 90.5
ERED	14°	90.4 91.8 91.8 91.8 91.8 91.8 91.8 91.8 91.8
IFFI	130	90.7 90.7 90.7 99.8 96.8 97.6 97.6 97.6 100.000.0
I QN	12°	91.1 92.5 93.5 93.5 93.6 93.6 93.6 93.6 93.6 100.0 100
A SI	110	91.4 92.4 94.8 94.8 94.8 94.8 94.8 94.8 94.8 94
SUN	100	91.7 98.1 98.2 99.5 99.5 99.5 99.5 99.5 100.5 100.5 100.5 100.5 100.5 111.2 100.5 111.5 11.5

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cent.	by vol. B	gram. 163.06 164.71 164.71 164.71 164.71 172.96 177.28 177.28 177.28 177.28 177.28 177.28 177.28 177.28 177.28 177.29 177.29 177.29 177.29 177.29 177.20 177
Per	by wt.	100 100 100 100 100 100 100 100
T.)	35°	255.2 127.5 127.5 127.5 129.0 129.0 129.0 129.0 129.0 144.2 155.8 144.2 155.8
CEN	340	2557 2557 2557 2557 2557 2557 2557 2557
ES (	330	$\begin{array}{c} 8.8.2\\ 8.$
TUR	50	2000 000 000 000 000 000 000 000 000 00
ERA	01	700001111311113111131111311113111131111
EMP	00 3	7.2.5.1.1.2.2.2.2.2.2.2.2.2.2.2.2.2.2.2.2
IE T	)c 3(	
IT 1	50	0.747 0.000 0.000 0.747 0.0000
U A'	28	
AKEI	270	1995. 1310. 1311. 1312.
E, T.	260	120.7 152.0 155.0 15
ERSI	250	2003 2004 2004 2004 2004 2004 2004 2004
INNI	240	30.5 33.0 33.0 33.0 33.0 33.0 33.0 33.0
QNN	230	33.21 33.21 33.51 35.11 35.11 35.11 35.11 35.11 35.11 35.11 44.1.5 44.5.757777777777
CT	520	33.71 33.71 33.72 33.72 33.72 33.72 33.72 33.72 33.72 55 55 55 55 55 55 55 55 55 55 55 55 55
DIRF	510	2322 11 12 23 23 23 23 23 23 23 23 23 23 23 23 23
NS, 1	000	22222222222222222222222222222222222222
TIO	90 2	28888881414920000011110000000000000000000000000
EVI/	80 1	51-10-20-20-20-20-20-20-20-20-20-20-20-20-20
ED	10	
TH	1	
s oi	16	411717171717171717171717171717171717171
NCE	150	
ERE	140	135.0 137.1 138.5 138.5 138.5 138.5 138.5 144.1 144.5 147.5
IFF	13c	$ \begin{array}{c} 138.5 \\ 138.5 \\ 138.5 \\ 138.5 \\ 144.6 \\ 144.6 \\ 144.6 \\ 144.6 \\ 144.5 \\ 144.5 \\ 144.5 \\ 144.5 \\ 144.5 \\ 155.6 $
I UN	120	126.6 138.4 1440.5 138.4 1440.5 138.4 144.3 144.
IS A)	110	173.2 177.2
SUN	100	1737 (1737) 1737 (

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ANALYTICAL TABLES.

# CHAPTER XXX.

#### ELECTRICITY.

SINCE the introduction and improvement of the many forms of apparatus now in use for the production of electricity and its kindred imponderables, it has become necessary for the chemist to be well acquainted with their mode of manufacture and employment. Many of the processes of the laboratory are now performed or assisted by means of them, and a knowledge of their construction is important not only as being the first step to an acquaintance with the principles concerned in their action, but also as affording the power of excelling in practical skill, and of preparing, extemporaneously, instruments to take the place of the more expensive ones made in the shops.

We propose accordingly to give an account of the mode of operation of the various instruments used for the production and detection of electricity, with directions for their proper employment, and some of their most common applications to the purposes of the experimenter and practical chemist, beginning with the one which is in most common use.

Cylinder Electrical Machine.-The various parts of this instrument are shown in Fig. 375. A, is a glass cylinder which is made to revolve by the turning of a winch connected by a crossed belt with the wheel and axle attached to the two axes of the cylinder. These turn in the supports seen at its sides. F, is the negative conductor supported by a glass pillar, and having on the side, nearest to the cylinder, the rubber or leather cushion which presses against its sides. The cushion has attached to it the silk flap G, which extends nearly over the upper part of the cylinder's circumference. Below, a screw passing through two nuts, is so placed as to increase or diminish the friction of the rubber upon the glass, by making the distance of the movable pedestal upon which the former stands, greater or less from the main support of the machine. Upon the opposite side of the cylinder, is the 27

positive or prime conductor C, being, like the other conductor, a hollow cylinder of brass or other metal, also insulated and



provided with balls for the transference of the influence, but having instead of a rubber, a collector E, provided with sharp points or prongs which are almost in contact with the surface of the cylinder.

The machine is often simplified by having the handle attached directly to the axis of the cylinder, and by dispensing with the screws and other parts of the arrangement in a way to be hereafter described. As arranged in the figure, it is made to develop electricity by turning the winch, which causes the smaller wheel and the attached cylinder to revolve with accelerated rapidity. By the friction of the glass upon the rubber-covered with a metallic amalgam-the electricity naturally present in the latter is supposed to be decomposed, its positive element becoming attached to the glass and its negative one to the cushion. The former, prevented from escaping by the non-conducting silk, is carried around with the glass, and-after a series of alterations by induction, of the equilibrium of that portion of the fluid naturally present in the conductor-finally by passing into it through the highly conducting collector, fills it with positive or vitreous electricity, which can be drawn off from it into other conducting bodies, either insensibly or in sparks. The negative conductor while insulated, becomes in the same way negatively electrified, and is capable of giving sparks to, or rather receiving them from, other bodies oppositely influenced: but while unconnected with the earth, it gives off to the cylinder but a small amount of its positive component, and it soon returns to its former state of equilibrium, from its negative electricity being neutralized by the positive kind of the prime conductor, which passes back over the surface of the cylinder, and which also reaches it by other sources of imperfect conduction.

If much positive excitement is desired, the conductor to which the rubber is attached, must be kept in connection with the earth, or a conducting surface in contact with it, by a metallic chain or wire. When, on the contrary, the intention is to obtain negative electricity from its proper collector, the prime conductor must be made to communicate with the earth by the same means.

Not a little care is necessary in the construction and arrangement of all the parts of the electrical machine. The cylinder should be strong and well annealed, particularly at the projections which are to be received into the caps forming the centres of motion. Its sides should be as straight as possible, so as to be adapted uniformly to the rubber, and to allow it to run truly upon its axis. Moisture should be carefully expelled from its interior, by a long-continued exposure to a current of dry and warm air, and the lateral orifices should be hermetically closed by cementation to the caps which form the axes. Cylinders of the proper size and form are sold in our glass-works and shops, but when one cannot be obtained, a properly made glass jar or bottle, may be so attached to a wooden or metallic axis-passing through its. mouth and through a hole in its concave bottom-as to make a very good substitute.

The cushion, or rubber, consists of a pad made of buckskin or soft leather, stuffed with horse-hair or similar material, and mounted upon a support which is in connection with the negative conductor. In the greater number of cases, there is no occasion for the presence of this latter part of the machine. When positive electricity alone is to be collected, the rubber may be with propriety attached to a wooden support, which is by any means made capable of being drawn towards, or separated from, the cylinder. The flap attached to the rubber is generally made of unoiled black silk, and it should reach nearly to the extremities of the pointed collector, so that its full effect may be produced, namely, the preventing of the transmission of electricity into the air before it reaches the prime conductor.

The exciting power of the rubber is much increased by spreading upon its surface an amalgam, mixed with unctuous matter, to make it adherent. A mixture of the amalgam of tin-scraped from the back of a mirror-with a little lard, answers the purpose very well; but the best kind, and that in most common use, is made by adding to six parts of mercury, previously heated in a crucible, a melted alloy of two parts of zinc and one part of tin, and by rapidly stirring the mixture until it is cold, when it can be readily reduced to powder. Before applying it, the cushion is cleaned and roughened by scraping, and is greased with a little lard or tallow. Some of the amalgam is then intimately mixed in a mortar with a quantity of unctuous matter sufficient to make it of a pasty consistence, and enough of this is smeared upon the surface of the rubber to give it a metallic appearance. The cylinder is then to be turned rapidly while the cushion is forcibly pressed upon it, and after the amalgamated surface has been compressed and equalized by the friction, the superabundance of grease and metal is wiped off from the surface of the cylinder and flap. The impregnation of the latter with a small portion of the amalgam, and the presence upon the surface of the former, of minute spots of it, are rather advantageous than otherwise. The mode in which the amalgam assists in the production of electricity is not precisely known, but it is supposed that its oxidation by friction and exposure to air has something to do with it. The facts that amalgams of gold and other difficultly oxidable metals do not increase the development of the power, and that an atmosphere of carbonic acid surrounding the machine prevents it entirely, seem to favor this belief.

The conductors of the two kinds of electrical influence are generally of the form shown in the figure, and usually consist of hollow cylinders of brass or tinned iron. They may be turned out of wood and covered smoothly with tin foil, which answers the same purpose, as it has been found that the electricity, even when in a state of great tension, is con-

#### THE PLATE ELECTRICAL MACHINE.

centrated chiefly in the surface. Small brass knobs or balls are attached by thick wires to the sides and tops of the conductors, for the purpose of drawing off sparks from the machine. In the absence of these, leaden bullets with wires inserted, may very well be used. The conductors are supported upon glass pillars or tubes, which are coated with a varnish of shell-lac. These, as well as the caps of the cylinder, are firmly inserted into their connections by means of the cement described upon page 276.

The Plate Electrical Machine.—The power of this description of machine is believed to be much greater than that of the kind in which the cylinder is used. The objection to its employment has been the difficulty of insulating the cushions, and consequently of obtaining negative electricity. It has, therefore, been chiefly employed for class demonstrations. Dr. Hare, in his Compendium, describes one which he has successfully used, a long time, for the purpose of producing both kinds of influence. The machine is represented in Figs. 376 and 377, and the following is the description:—

"The plate B (thirty-five inches in diameter) is supported, as represented in the figure, upon an upright iron bar, about an inch in diameter, covered by a very stout glass cylinder A, four inches and a half in diameter, and sixteen inches in height, open only at the base, through which the bar is introduced, so as to form its axis. The summit of the bar is furnished with a block of wood, turned to fit the cavity, formed at the apex of the cylinder, and cemented therein. The external apex of the cylinder is cemented into a brass cap, which carries the plate. The glass cylinder is liable to no strain. It is only pressed where it is interposed between the block of wood within, and the brass cap without. The remaining portion of the cylinder bears only its own weight, while it effectually insulates the plate from the iron axis. The brass cap is surmounted by a screw and flange, by means of which, a corresponding nut, and disks of mahogany, the plate is fastened. A square table serves as a basis for the whole. The iron axis, descending through the top of the table, is furnished with a wooden wheel of about twenty inches in diameter D, (Fig. 377,) and terminates below this wheel in a brass step S, supported on the cross of wood, which ties the legs of the table diagonally together. The wheel D, is grooved and made to revolve by a band, which proceeds from around a

# THE PLATE ELECTRICAL MACHINE.

vertical wheel W, outside of the table. This external wheel has two handles, by means either of one or both of which it



may be turned. It is supported on two strips of wood G G, which, by appropriate screws (represented at S S, Fig. 377),

Fig. 377.


may be protruded, lengthwise, from cases, which confine them from moving in any other direction. Consequently, the distance between the wheels may be varied at pleasure, and the tension of the band adjusted.

"Nearly the same mode of insulation and support, which is used for the plate, is used in the case of the conductors. These consist, severally, of arched tubes of brass, of about an inch and a quarter in diameter, which pass over the plate from one side of it to the other, so as to be at right angles to, and at a due distance from, each other. They are terminated by brass balls and caps, which last are cemented on glass cylinders C C C C, of the same dimensions, nearly, as that which supports the plate. The glass cylinders are suspended upon wooden axes, surmounted by plugs of cork, turned accurately to fit the space which they occupy. The cylinders are surrounded and secured below, by wooden rings screwed to the table. In this way the conductors are effectually insulated, while the principal strain is borne by the wooden axes.

"Collectors consist of hollow hemispheres of sheet brass, within which several points proceed towards the plate from their centres respectively, where they are attached to the knobs K K K K. The hemispheres are intended to diminish the injurious circulation of air.

"The cushions are included between springs, by which they are made to press with an elastic force upon the surfaces of the glass, the degree of the pressure being regulated by a screw."

Electrical machines of whatever kind, act to the greatest advantage in clear, cold and dry weather. A moist and warm condition of the atmosphere is generally unfavorable to their use, but various circumstances,—of which unknown meteorological influences are probably the chief—oppose the electric excitement even in an apparently propitious state of the weather. Care should be taken in such cases, whatever the condition of the air, that the communication of certain parts of the apparatus with the earth be as perfect as possible. With this view, when either kind of excitement is required, the conductor of the other kind may be connected by means of a chain or wire with the water or gas pipes of a house, or any other good conducting substances which penetrate the moist earth. The cylinder, or plate, and the insulating parts of the apparatus should be free from moisture or dust, which might both conduct away electricity, and to ensure perfect dryness of all parts of the machine, it should be placed before a fire or upon a stove or sand bath, and be thoroughly rubbed and dried with a piece of warm flannel, taking care that the amount of heat applied be not great enough to melt the cement, which is employed to connect the various parts.

It is an indication that the machine works properly, if after several revolutions of the cylinder or plate, the approach of a metallic ball or of the knuckle to a part of the insulated conductor, causes a vivid spark to dart with a crackling noise from the latter to the former. The size of the spark and loudness of the report are of course dependent in a measure upon the magnitude and power of the machine.

The Leyden Jar.-This instrument, shown in Fig. 378, is

Fig. 378.



the one employed for the purpose of accumulating electricity by *induction*, and is the agent chiefly made use of for laboratory purposes. It usually consists of a wide-mouthed jar of thin glass, coated with tin foil both upon the inner and the outer side of the bottom and the lower two-thirds of the circumference. The stopper is made of cork or dry wood, well varnished and cemented tightly in its place. Through its centre, a wire or rod passes, which, either with or without a chain attached, is in close contact below with the inner lining of the jar, and which terminates above in a smooth metallic

ball. That part of the glass which is not covered with tin foil is usually painted over with a coating of shell-lac or common spirit varnish. The jar made use of in the laboratory, need not ordinarily exceed a quart in capacity. The ball upon the top of the rod should be at least an inch in diameter, and the latter should be so firmly fixed in its place as not to be dislodged from its connection with the stopper or the bottom of the jar, by any change of position.

A common phial containing iron filings—into which is plunged a wire which passes through the cork, and is inserted into a bullet above—and coated outside with tin foil up to the level of the metal within, makes a very efficient apparatus.

The Leyden Phial is charged by placing its ball in contact with that conductor of the machine which contains the kind of electricity which it is intended to accumulate in its inner coating, while the outer metallic surface is in connection with the earth. This is most conveniently performed by grasping the jar in the hand, and presenting the knob to the conductor during the continued turning of the winch.

The electricity often accumulates in the inner coating until its tension becomes so great that the equilibrium of both coatings is restored by a discharge taking place from one surface to the other; that amount which is in excess leaping over, as it were, the intervening non-conducting surface of glass. When one kind of electricity is collected in the interior, the opposite sort is produced in the exterior coating, or-in accordance with the theory which admits the existence of only one kindwhen it is in excess in the inside, it is deficient in the same ratio upon the outside. The tendency is then always to restore that neutrality, or natural order of things in which the influence is not made evident to the senses; but in a good Leyden jar, the parts should be so arranged as to permit the collection of a large amount of electricity before a spontaneous discharge takes place. If the jar, before becoming highly charged, permits such an escape, it is usually an evidence that there is a hole or crack in the glass, that the uncovered surface has become a partly conducting one from the presence upon it of moisture or dust, or finally that the outer metallic coating extends up so far as to allow of the easy passage of a spark to it from the rod or ball. The last condition can be altered by lessening the height of the outer covering of foil, taking care to reduce that which is within, also to the same level. To expel moisture and dust, the vessel should be warmed and wiped, as before directed for other parts of the apparatus.

The retention for some time, of the charge, by the Leyden jar is often a matter of great importance in the chemical applications of electricity. A jar, of the capacity above mentioned, when dry, warm, and fully charged, should after a lapse of ten minutes, give a spark at least half an inch in length, to the ball of a discharging rod, the ball being onethird of an inch in diameter.

The power of the jar is dependent on the amount of the coated surface, and the thinness of the glass.

The Electrical Battery.—This is an arrangement by which the metallic surfaces of the Leyden jar are greatly increased in size, and by which the intensity of the shock and discharge is multiplied to almost any extent desired. A number of Leyden jars prepared in the usual manner, are placed in a box which is lined with tin foil or other metallic coating. The vessels are placed in close contact, or are made to connect with each other externally, by the interposition of metallic or coated partitions, and the inner coatings are made to communicate by means of metallic rods or chains, connected with the wires going from their interior. The whole is equivalent to a single large jar, and may be charged and discharged with equal facility. Fig. 379.

# Fig. 379.

The hook, seen in the front of the box which contains the series, is attached to the metallic outer lining.

When the battery is to be used, it should be ascertained that all the outside coatings are in proper connection with each other, and that the inner surfaces communicate through their appropriate mountings and wires, and that no wires, threads, water, or other conducting substances extend in any way from the inner to the outer parts of the apparatus. No filamentous or pointed body, or projecting piece of metal, should be allowed to remain very near the battery while it is in operation. The battery is charged by connecting one of the wires or knobs which are in contact with the inner coating of the jars, by means of a chain or wire, with the prime or negative conductor of the electrical machine while it is in operation. It is both filled and discharged in the same way as the Leyden jar, and all its operations are those of that vessel upon a greater scale.

During the charging of a battery, a diffusion of electricity

### THE DISCHARGER.

sometimes takes place over that part of the uncoated glass, which is near the edge of the foil. This is not entirely removed upon the discharge of the coated part, but afterwards gradually returns to the coating and recharges the battery, often to a considerable extent. Hence if after the discharge of a battery, it be left for a few minutes with the two coatings unconnected, it will, upon the application of the discharger, give a considerable spark. This, which is the *residual charge*, is discharged in the same way, when the Leyden jar is used.

The Discharger.—A discharge between the oppositely electrified surfaces of the jar may be effected by bringing one hand in contact with the external coating, and touching the knob with a knuckle of the other. In this case the person receives a shock in his arms, and if the surfaces are large or well filled with electricity, he experiences a painful passage of this shock through the shoulders and chest. A battery ordinarily charged, should never be discharged in this way, as serious and even fatal results might follow.

The instrument, shown in the figure, is called a discharger and is used to complete the circuit between the opposite coat-



ings of both the jar and the battery. The rods R, R, are so united with a hinge that the balls may be made to come in

contact with the surfaces, or to be removed from them by means of the insulating glass handles, which are attached to the legs. This arrangement gives the power of discharging a jar of almost any size, by removing or approximating the handles. A very effectual and cheap discharger is made of a piece of thick wire, about twelve inches long, curved and terminated by a bullet at each end.

### OTHER MEANS OF PRODUCING ELECTRICITY.

The Electrophorus.—This important instrument, Figure 381, may in many cases be made to take the place of the



common electrical machine. A mixture of equal parts of common resin, shellac and Venice turpentine, is melted and kept in a state of fusion at a temperature between 230° and 240° of Fahrenheit, until nearly all evolution of vapor has ceased and the fluid is quiet. It is allowed to cool to the point of thickening, and is then poured care-

fully, so as to avoid the formation of air bubbles, into a circular metallic tray or dish, of about nine or twelve inches in diameter, and half an inch in depth. The resinous surface should be as even and smooth as possible. A wooden box, or a receptacle made by placing upon a smooth board a wooden hoop, are less costly and do not expose the resin to the risk of cracking from the sudden contraction of the metal, which is apt to occur after its expansion by heat. Upon the smooth surface formed by the cooled mixture, is placed a metallic disk, or one of wood smoothly covered with tin foil, either of which is provided with an insulating handle of glass or sealing wax, which is inserted in its centre; above. This disk should be somewhat less in diameter than the surface of resin. The top has usually attached near its edge, a wire terminating in a metallic ball, from which the spark is taken.

When this electrophorus is used, the cover is removed, and the surface of the resin having been dried and slightly warmed, is rubbed or whipped briskly with a piece of dry flannel, a silk handkerchief, or the fur of a cat or hare's foot.

# HENLEY'S QUADRANT ELECTROMETER.

After excitation by this means, the cover is lifted by its handle—also dry—and is replaced upon the surface of the resin. A spark will now pass from the knob of the cover to the knuckle, or a metallic body held near it. Upon raising the cover again, another spark of greater intensity than the first will be received. A spark like the first, will be given by the knob after the replacing of the cover, and again upon its withdrawal, one similar in character to the second will be given off, and in this way the experiment may be repeated almost indefinitely if the weather is favorable.

The action of the machine is explained in this manner. The negatively excited cake of resin acts inductively upon the electricity inherent in the cover, attracting and combining with its positive element, and repelling its negative one, which accumulates in the upper part of the cover. When the top of the cover is touched, the negative electricity escapes, and the positive remains in combination with the negative kind of the resin, as long as the latter is covered by the metallic plate. But upon lifting this by the insulating handle, the positive excitement is in its turn set free, and given off in sparks from the knob. A similar succession of actions goes on for some time, and the instrument has been known to give sparks for weeks without being freshly excited.

To obtain strong positive sparks, it is necessary to touch the cover when on the resin, with a finger or other conducting body, and to remove it before raising the cover. To obtain the strongest negative sparks, the cover when raised, should be discharged of all its electricity against the hand or other body before it is again placed upon the surface of the resin.

# INSTRUMENTS FOR DETECTING AND MEASURING ELECTRICITY.

Henley's Quadrant Electrometer.—This instrument, chiefly used to determine the amount of electricity present in the conductor and in the Leyden jar, consists of a semicircle of ivory or of wood covered with white paper, which is graduated into 180 degrees, and fixed at its base to a wooden column. In the centre of the semicircle there is a pin upon the column, from which a movable radius terminated by a pith ball is suspended. The column may be fixed in a hole in the conductor. Upon working the machine, the column and the ball being

alike affected, the latter with its radius is repelled from the former, and by the amount of the divergence the force is exhibited in degrees. By means of this instrument, we are enabled to ascertain when a jar, or battery in contact with the conductor, is sufficiently electrified. During the accumulation in the inner coating, the electricity is retained forcibly by the attraction of the contiguous and oppositely electrified surface, and will not be given off to an insulated body, or one which is not in connection with the outer coating. But in proportion as it ceases to be retained by this inductive action; and accumulates in the conductor, it raises the index of the electrometer, often to a considerable height. When a battery has received its greatest amount of charge, the ball seldom rises above 40° or 50°, as the tension of the electricity never equals that of a single jar, probably on account of the larger surface exposed to induction.

Haüy's Electroscope has already been described under the head of the Blowpipe at page 383.

Bennet's Electrometer.—This instrument, more properly called an electroscope, as it detects, rather than measures electricity, is exceedingly delicate in its indications. It consists in part of a glass cylinder, which may be similar in form to the one shown in the drawing. A circular brass cap C,





covers tightly the vessel, and to its centre is attached a metallic rod, enclosed in a glass tube which is well varnished with shell-lac, and having attached to its ends two slender strips of gold leaf, hanging parallel to each other. Two strips of tin foil T T, are pasted upon the inside of the glass, with their upper ends a little above the level of the depending extremities of gold leaf, and their lower ends connected with the metallic bottom of the glass cylinder. When an electrified body is made to approach the cap of the electrometer, the gold leaves will diverge, and if the excitement be

sufficiently powerful, will touch the tin foil and then return to their former state of rest.

# BENNET'S ELECTROMETER.

The delicacy of Bennet's electrometer is much increased by the addition of two metallic disks, one having its centre soldered to the side of the cap of the instrument, being in a perpendicular position, and the other being attached to a rod which is connected with the metallic foot of the instrument by a hinge, so that it may be placed parallel to the other disk, and so near as almost to touch it without actually doing The presence of electricity in the metallic cap, and its SO. disk, induces the opposite kind in the contiguous metal, which is then to be removed a few inches from its former position. As this disk is connected with the base of the instrument, and of course with the tin foil upon the inside of the glass, that becomes also oppositely electrified from the cap, the connected gold leaves of which diverge to a much greater degree than in the simple instrument. This is called the condensing electrometer.

Bennet's electrometer is the one in most common use, and many circumstances of interest in reference to its employment are worthy of note, particularly those connected with the means of ascertaining the kind of electricity which causes its gold leaves to diverge.

If an insulated conducting body containing electricity, such as the prime conductor of the electrical machine, is made to approach or to touch the cap of the electrometer, the leaves diverge to a greater or less degree, in proportion to the tension of the electricity in the body, and remain separated, gradually returning to their former position as the influence passes off. In examining the condition of a body supposed to be highly electrified, care must be taken not to make it approach the cap too rapidly, as the result of a sudden and powerful communication of the agent is very often the immediate separation and tearing of the gold leaves. When a non-conducting body, electrically excited, -a piece of sealing wax, for instance,-is brought near to, or in contact with the top of the instrument, the same divergence takes place; but it is temporary, as upon the withdrawal of the body the leaves come together again. To make their separation as lasting as in the former case, it is necessary either to allow the body to remain for a time upon the cap, or to rub it over its surface, so that it may communicate its electricity from a number of points. So far, the electricity of either kind, imparted to the cap, has been that of conduction. But if the electrified

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body be held so near to the cap, as just to cause the divergence of the leaves, that divergence will diminish gradually until the leaves finally collapse. If now the body be removed to such a distance that it can scarcely affect the leaves, they, after coming together, will often gradually diverge as before. This second separation is caused by *induction*, and when it occurs, the opposite kind of electricity to that existing in the body will be found to be present in the cap and leaves. The same effect is produced by touching the cap with the hand, while the leaves are diverging from the electricity of the excited body, by removing the hand after the collapse occasioned by its first contact, and by then withdrawing the electrified body, as before, to a greater distance from the cap.

It is well known that a piece of sealing wax, rubbed with warm flannel, becomes negatively electrified and that a glass tube rubbed with a silk handkerchief, becomes positively affected. These facts present us with the means of determining the kind of electricity which is transferred to the cap and leaves of Bennet's electroscope. If—after the leaves have been made to diverge by the approximation to the cap of an excited body—the presence upon the top, of a piece of rubbed sealing wax makes the divergence greater, the electricity in the body is negative. If however, the leaves approach each other slowly, or collapse at once, the electricity is more or less positive. In the same way, a warm tube of glass, rubbed with a silk handkerchief, will increase the separation of the positively electrified leaves, and diminish or annul it when they are negatively excited.

Coulomb's Electrometer.—All the instruments, above described, indicate the presence of electricity, but give little idea of its quantity. Coulomb's torsion balance gives us an approximation at least to a means of accurately measuring it, or rather of comparing the amount of it found in one body with that existing in others, or in the same body, at different times. This instrument, as represented in Fig. 383, from Golding Bird's Natural Philosophy, consists of a slender beam, or thread of shell-lac B, having a gilt pith-ball attached to one end, and a little vane of paper to the other, and suspended at its centre by a fine metallic wire, or what is better, a delicate filament of spun glass. This ascends in a cylindrical or square frame of glass, and its upper end terminates

## COULOMB'S ELECTROMETER.

in a key D, furnished with an index, the whole being capable

of moving easily in the centre of the circle G, which is graduated into 360°. A rod of shell-lac F, is inserted in the hole E, and is prevented from falling down into the glass cylinder which surrounds the whole arrangement, by a stop at E. This rod terminates in a gilded ball, which is called the carrier ball, as it is used to convey to the electrometer proper, the electricity of the excited body. When this instrument is to be used, the rod F is brought into contact with the excited body; its ball acquires some of the electricity, and upon being placed in the cage, it gives a part of it to the ball of the lac beam. This having now the same kind of electricity, is repelled from the ball of the rod and describes a certain angle to its former posi-



tion, which it retains until it loses its electricity. To measure the amount of fluid thus acquired, the key D, to which the glass thread is fastened, must be turned around, until by the torsion or twisting of the latter, the ball of B is made to come in contact with that of F. The number of degrees described by the index, which is attached to the revolving key D, gives an approximation to the proportion of electricity derived from the contact of the ball of F with the electrified body.

A more simple form of this electronometer, and the one ordinarily described, consists of a lac needle with a gilt ball at each end, suspended by means of a fine untwisted thread of raw silk, which is fixed at top to a micrometer, by means of which it can be turned around any number of degrees required. The whole is encased in a glass vase or cylinder, with a tightly fitting top of glass, through a hole in the centre of which the silk passes, the micrometer being above. Upon the level of the suspended needle, a hole, drilled through the sides of the glass, encloses a wire having a metallic ball at either end, the inner one being nearly in contact with one of the pith balls. The excited body is made to approach the outer ball, and as in the instrument before described, the movable knob separates from the other, and the quantity 28

### EUDIOMETRY.

of electricity is proportional to the distance to which it is driven off.

### APPLICATIONS OF ELECTRICITY.

Eudiometry .-- Electricity proper is more often applied in the laboratory of the chemist to the analysis of gaseous mixtures, by taking advantage of its power of exploding certain of these, than to any other purposes. It is employed in connection with the eudiometer, an instrument which is used chiefly, as its name indicates, to ascertain the purity of the atmosphere, but in which the analysis of gases containing carbon and hydrogen is also occasionally effected. In it, these latter are made to unite explosively with oxygen, while in the examination of the atmosphere, the explosion of hydrogen with its constituent oxygen, and the consequent production of water and diminution of volume, enable the chemist to determine the proportion of its ingredients. It would be foreign to our purpose to give a full description of all the applications of eudiometry, but a short account of the means most commonly employed, particularly in reference to the proper mode of applying the electric spark, will scarcely be at variance with the practical nature of this work.

The Common Eucliometer is a short tube of thick glass, having one end closed. This tube is graduated, and near its closed extremity, two stout wires of platinum or other metal, intended for the transmission of the spark, are inserted in the opposite sides, their ends inside of the tube being a short distance apart. The other end of the tube serves for the introduction and escape of the gas, and it remains constantly immersed in the liquid over which the experiment is made, the tube being supported in a perpendicular position. The gas to be subjected to the spark, is generally such a mixture as will inflame explosively at once, though sometimes a gradual combination of some of its elements is effected by means of a long-continued succession of sparks. The tube, being filled with water or mercury, may be placed over the trough; or for the purpose of more accurately determining the level of the gas in the way about to be described, it should be supported over a glass vessel containing the proper liquid. The gases are then successively introduced into it, in the proper

### THE COMMON EUDIOMETER.

proportions, after the manner described upon pages 134, 135. To determine their volumes with the utmost degree of accuracy, it is necessary to support the tube by a forceps or a cork-lined clamp, as represented in the figure, and not be-



tween the fingers, so that their temperature and volume shall not be increased by the heat of the hand. To ensure that the gas be submitted to no more pressure than that of the atmosphere, the eudiometer should be raised in such a manner that the interior level of the liquid contained in it, shall be exactly at the same height as that of the liquid in the vessel outside. In order to secure this, it is necessary that the eye of the observer be in the same plane as the two levels of the liquid, and that the line of the liquids in direct contact with the glass inside and outside of the tube, be not taken as the proper standard. It must be recollected that-as the edge of a surface of water, in contact with the glass, is elevated above its true level by capillarity, and that of mercury in the same circumstances is depressed-the lower line in the former case, and the upper one in the latter, will give the true position of the main surfaces. The exterior of the tube is now wiped clean, so that no mercury or water in contact with the wires, can conduct off the electricity. The tube, kept upright, should then be clasped firmly in the hand by its middle, and its lower end, still under water, should be closed with slight force by

### THE EUDIOMETER.

the thumb or a finger of the unoccupied hand. This permits the descent of the fluid, which is driven out by the force of the explosion, while it does not allow its too sudden return upon the subsequent contraction of the gaseous contents of the tube, or the escape of any of the latter.

In using the eudiometer, we must take into account the relative degree of explosibility of different mixtures. Thus a mixture of oxygen and carbonic oxide expands when inflamed, much less than one of oxygen and hydrogen or olefiant gas. A large quantity of any mixture will of course increase in bulk much more than a small one. The whole quantity of gas contained at first in the tube, should be at least so small, that after expansion it shall not occupy quite the whole of the eudiometer. No more gas should be introduced for detonation than will occupy a sixth of its capacity at common temperatures, and generally it will be advisable to employ much less.

The spark which is intended to effect the detonation or slow union of the gases contained in the tube, may be derived from the electrophorus, the prime conductor of the electrical machine, or the Leyden jar, the power of the last two being of course greater, in the order in which we have spoken of them, than that of the first. When the electrophorus is employed, one of the wires upon the side of the eudiometer is placed in connection with a finger of an assistant, or with a metallic chain, the other end of which hangs in the trough or vessel over which the tube is supported. The ball of an excited electrophorus is then brought near to the other wire, and the spark obtained from it, passing from wire to wire through the interior of the tube, inflames the mixture, if it be of sufficient intensity, and if all the other circumstances are favorable. The ball upon the conductor of the electrical machine may in the same way be made to approach one of the wires, with usually a more powerful effect. The employment of the electrical machine is particularly advantageous when it is desired to pass a succession of sparks for a considerable time through the mixture, for the purpose of effecting a gradual combustion or combination of the gases contained in the tube. The use of the Leyden jar is equally convenient for a single contact and much more apt to be attended with success on account of the greater size and force of the spark. One of the wires may be connected with the external coating of the jar, by means of a

chain or hooked wire, and a discharger or other conductor, applied at one end to the ball of the phial, may be brought near the other wire. When other means of connection are not at hand, the operator, at the risk of receiving an unpleasant shock, may grasp the jar in his hand and apply its ball to one wire of the eudiometer, while he touches a finger of the other hand to the opposite wire. To ensure the explosion of the mixture, a spark of the largest size that can be obtained from the electrical instrument, should be passed through it. Very often, although a sufficient amount of electricity is given off from the conductor of the electrophorus or electrical machine, its effect is lessened by its communication from wire to wire, as an electrical brush, or in a succession of small sparks. To remedy this evil, a ball, half an inch or more in diameter, should be placed upon the outer extremity of that wire which is to receive the spark, and the latter should always be given off from the surface of a ball of considerable size.

The wires of the eudiometer must be firmly fitted in their places, and the openings in the glass through which they enter should be hermetically closed around them. Before filling the tube with gas, it must also be ascertained that they are perfectly insulated. When the detonation is effected over water, a film of it is apt to adhere to the glass and wires, both internally and externally, which by its conducting power, sometimes diminishes the force of the spark, or intercepts it entirely. To prevent this, the outside of the tube and wires must be wiped as dry as possible before applying the conductor. The top of the tube should be gently tapped so as to shake off any particles of moisture adhering to it within. The perfect transmission of a large spark is only secured by the presence of the balls upon the ends of the wire and discharger as before described.

Ure's Eudiometer.—Analysis of gases by explosion is much more conveniently performed by means of Dr. Ure's syphon eudiometer, shown in Fig. 385. It differs from the other eudiometer in being curved like the letter U, but like it, it has the part intended to contain the gaseous mixture, graduated and pierced by two platinum wires. It is usually about twenty inches in length, and the third of an inch in internal diameter. This instrument, like the other, may be used for the analysis of various gases over either water or mercury,

# URE'S EUDIOMETER.

but as it is applied chiefly to that of atmospheric air over the



latter liquid, we will confine ourselves to a short account of this employment of it. When about to be used for an examination of the atmosphere, it is filled with mercury, and the required amount of air is introduced into the open end, which is inverted over the trough, as in the case of the use of the other form of tube. This end is then tightly closed with the finger, and the tube is turned slowly so as to admit the air into the graduated extremity. The instrument is then held upright, and the amount of air introduced is read off by looking at the scale, after subjecting it to atmospheric pressure by displacing, with a stick thrust in, that portion of mercury

which is above the level of that in the graduated limb. This having been accurately done, the open part is again filled with mercury, closed with the finger, inverted into the liquid, and an amount of pure hydrogen is introduced equal as nearly as can be guessed to half the volume of the air. The quantity of hydrogen added is then accurately estimated by returning the eudiometer to the erect position, equalizing the surface of the mercury as before, and reading off its level. The instrument is then held in the way represented in the figure, the thumb firmly closing its aperture, and the knuckle of the fore-finger touching the nearer platinum wire. The explosion is produced by the aid of the electrophorus, prime conductor, or charged jar, as before described, the violence of the expansion being moderated by the spring-like action of the air contained in the open limb. The level of the mercury is again equalized by pouring into the open side enough of it to produce that result, and the volume of the gaseous mixture is then finally read off.

The loss in volume of the mixture, which is produced by the explosion, gives by a very simple process, the amount of oxygen originally contained in the air. As hydrogen unites with oxygen to form water in the proportion by measure of two to one, one-third of the diminution must be due to the oxygen of the air introduced. Thus, if 100 measures of air and 50 of hydrogen have been introduced, and if the mixture contain

### GALVANISM.

only 87 measures after explosion, the diminution has been that of 63 measures. One-third of this loss is equal to 21 measures, which represents the amount of oxygen in the 100 measures of the air first introduced.

The precautions spoken of in reference to the common eudiometer may with equal propriety be applied to this.

### GALVANISM.

All the forms of apparatus which are employed for the purpose of producing a continuous electrical current, are called galvanic circuits, and those in common use consist of two metals, one more oxidable than the other, and of a liquid which by its action upon the readily oxidized or active metal, causes the development of the influence. The old voltaic pile and the crown of cups are the most simple examples of galvanic apparatus. The former consists of a series of disks of zinc, and copper, platinum or silver, arranged in a column, each piece of different metal having placed between it and its neighbor, a disk of cloth or paper steeped in some liquid, which acts chemically upon the zinc. The crown of cups is differently arranged, but upon the same principle. A number of cups are placed in a row or circle, each one containing an exciting liquid, such as dilute sulphuric acid, and a plate of zinc, and one of the inactive metal. The zinc of one cup is connected by a wire with the copper or other metal of the next cup, and the zinc of that is also connected with the copper of the one beyond it. The two external plates of both kinds of series have wires soldered to them, which are called the poles. In this way a communication exists between all the parts of the series, directly between the alternate plates of the different cups, and indirectly through the liquid between those in the same cup. A simple circuit, as exhibited by the most elementary form of either of these arrangements. represents in miniature all the other kinds of voltaic apparatus employed. Thus, if a single cup be used, containing a plate of zinc and one of copper, immersed in dilute acid, and having wires attached to them, the voltaic current is supposed to be developed upon the surface of the zinc, along with its partial solution and the evolution of hydrogen, to pass through the liquid to the copper, and to be conducted through that

metal to the end of its wire, which forms the *anode* or positive pole. The end of the wire attached to the zinc is the *kathode* or negative pole. When these poles are placed in contact with each other, or with a conductor of the fluid, the electricity originally developed upon the surface of the zinc returns to it from the positive wire through the negative one, and if the current be sufficiently powerful, the various phenomena of voltaic light, heat, electro-magnetism, chemical decomposition and action on the living body, are capable of being exhibited during this passage from pole to pole.

In most of the forms of compound circuits, where a number of pairs of plates are arranged together in a battery, the wire attached to the terminal zinc plate becomes the positive pole, and that of the last copper plate the negative one. This arises from the fact that in these arrangements the last two plates are actually superfluous, not being so much producers of the galvanic fluid, as conductors of that which has been generated in the intermediate parts of the apparatus.

Our limits will scarcely permit a reference even, to very many of either the theoretical or practical points connected with the phenomena of this extensive subject, nor does it come precisely within our province to notice the former at all. We will therefore confine our attention to the construction and uses of the forms of voltaic apparatus, which are the best known and the most used in the laboratory of the chemist.



Wollaston's Battery .- This is the very best of the old

forms of the voltaic battery. It consists, as shown in Fig. 386, of a number of zinc and copper plates, the latter entirely encircling the former except at the edges, and the two metals being kept apart by pieces of cork or wood. Each plate of zinc is soldered to the one of copper which is before it in the series, and the whole arrangement is screwed to a bar of dry mahogany, which permits its elevation from or depression into the acid. This is contained in an earthenware trough, divided by partitions into compartments, each one of which receives a single pair. The exciting liquid is made of a mixture of 100 parts by measure of water,  $2\frac{1}{2}$  parts of sulphuric acid, and 2 parts of strong nitric acid. In the same manner that the shock of the Leyden jar is increased by combining it with others in a battery, the power of this apparatus can be multiplied to any desired extent, by uniting it by means of strips of copper, passing from the zinc of one instrument to the copper of another, with any desired number of similar batteries.

The chief objection to the use of this and like forms of apparatus is what is called the *local action*, which in it is very great, and which gives rise to a rapid diminution of power and corrosion of the zinc.

The bubbles of hydrogen given off from its surface, adhere to the zinc, preventing perfect contact with the exciting fluid; some of the electricity is dissipated by the escaping gas, and the sulphate of zinc which is formed, is in part reduced to the metallic state, in a crust upon the surface of the copper. All of these circumstances form serious objections to the use of this battery where a long continued action is desired.

When common zinc is exposed to dilute sulphuric acid, it is rapidly dissolved, and this solution and loss of material in the common batteries are excessive and entirely disproportional to the amount of galvanic fluid given off. This, which is the *local action*, is supposed to arise from a number of little voltaic circles being formed by the presence in the zinc of particles of plumbago, and of other metals which excite the rapid erosion of parts of its surface. This evil can only be prevented by carefully amalgamating the surfaces of the zinc plate.

A single pair of Wollaston's battery is very efficient in the production of the phenomena which are due to the evolution of a *quantity* of electricity, such as ignition and deflagration on a small scale, the deflection of the magnetic needle and the various electro-magnetic experiments. Its *intensity* or electro-chemical power is very much increased as before stated, by combining it with other similar arrangements.

The plates of the old form of voltaic apparatus should be removed from the acid, and washed with water after the completion of each experiment, or if they are permanently connected with the trough, the acid in it should be poured out, and reserved for future operations. In Wollaston's battery, the plates are taken out by elevating the mahogany bar to which they are attached, are freed from acid and metallic deposit by washing with water, and are then either suspended over the trough by a cord attached to a support above, or are placed upon a tile or old table until their next employment. As the acid solution soon becomes unfit for use from the large amount of sulphate of zinc dissolved in it, it must be removed after reaching a certain point of saturation. The best evidences of the cleanliness and perfect connection of the surfaces, and of the activity of the liquor, are afforded by the constant bubbling up of hydrogen during the action, and by the ordinary voltaic phenomena exhibited at the poles.

Daniell's Constant Battery.—This is a far better form of apparatus than the one last described, and has the advantage over it of being comparatively permanent in its action. The local action being obviated by the amalgamation of the zinc, it is of course much more applicable to those purposes of electro-chemical examination in which long continued and uniform transmission of the fluid through a body is desired.

Fig. 387.

In its simplest form, it consists of a copper cylinder A, 3 or 4 inches in diameter, and from 6 to 18 inches in height, containing in its interior a cell of porous earthenware or of animal membrane, within which is suspended a rod of zinc three-quarters of an inch in diameter, which has been carefully amalgamated by rubbing its surface with mercury by means of a cloth previously dipped in dilute sulphuric acid. The cell or membrane containing the zinc is filled with a mixture of one part by measure of sulphuric

acid and 8 parts of water, and the space between it and the

outer copper cylinder contains a saturated solution of sulphate of copper, the surface of which should be upon the same level as that of the solution within the cell. The solution of blue vitriol is prepared by pouring boiling water over an excess of crystals of the salt, and stirring constantly until it is saturated.

To this solution, a little sulphuric acid, never amounting to more than one-tenth part by measure, of the whole, should be added. In order that this liquid be kept concentrated, a little perforated copper shelf, seen in the figure, is usually placed upon the inside of the cylinder, within an inch or two of the top. This is intended to contain a supply of crystals of the sulphate. They are placed at the upper part of the liquid, because that portion becomes exhausted first, and because the saturated solution of the crystals in its passage downwards diffuses itself equably. In the absence of the shelf, a strong bag of loose texture, or a net-work of copper wire attached to the top of the cylinder, may be used to contain the crystals.

Attached to each metal of Daniell's battery is a binding screw to form connections. When wires are held in each of these, and a communication from the cylinder to the rod is made, a powerful current is produced. In the figure, the extremity of z represents the positive pole, and that of x the negative one. In this arrangement there is no evolution of hydrogen, and no local action upon the zinc or consequent unnecessary erosion of its surface. The interior of the copper cylinder becomes covered with a compact deposit of metallic copper from the decomposition of the oxide by the nascent hydrogen.

The *intensity* or power of producing electro-chemical decompositions of this battery, may be much increased by associating it with a number of others. Ten pairs, so arranged that the inactive metal of one is attached by copper wires or strips, to the active metal or the zinc of the next, make a most powerful compound circuit, quite sufficient for nearly all the purposes of the chemist.

Daniell's battery may be constructed very simply and cheaply, by immersing in a tumbler or jar containing a solution of sulphate of copper, a copper plate of the proper size, bent into the form of a cylinder, and having suspended in its centre upon a piece of wood supported on the top of the outer vessel—an amalgamated zinc bar. This is surrounded by a piece of bladder or of the intestine of an animal, tied at its lower part, and containing the acid liquor. Bags of very firm sail cloth, well sewn, make excellent diaphragms and resist the action of the acid for a long time. Cylinders made by cementing coarse strong brown paper, at the edges and bottom, also answer perfectly well. The terminal wires may be soldered upon the top of the metals with which they are to be connected, and the solution of sulphate of copper may be kept saturated by the means before spoken of. Very little chemical action upon the surfaces of these batteries goes on when the voltaic circuit is not completed: nevertheless it is proper always to pour out the contents of the diaphragm or to disconnect the zinc bar after each use of them. The liquid may be kept in a separate vessel, and employed in future experiments. The solution in the outer cylinder may be allowed to remain.

Smee's Battery.—This simple and powerful apparatus is chiefly used to excite the precipitations of metals in the *Electrotype* or galvano-plastic processes. As commonly constructed and shown in Fig. 388, it consists of two plates of amalga-

Fig. 388.

means of a bent strip of brass, and furnished with a binding screw. Between the plates of zinc, is fixed one of platinized silver, connected at its upper end with another similar screw. The silver is covered over with a thin layer of platinum, by first roughening the surface with strong nitric acid, and after washing, placing it in a vessel of water acidulated with sulphuric acid, to which a little chloride of platinum has been added. A porous vessel of pipeclay or earthenware, or an animal mem-

mated zinc, clamped to a piece of wood by

brane, with a plate of zinc in its interior, and containing dilute sulphuric acid, is then immersed in the other receptacle, and the silver and zinc are connected together by a wire. The platinum precipitates upon the silver surface as a dark and granular but closely attached deposite.

This rough surface of the silver plate, presenting myriads of minute conducting points, greatly facilitates the evolution of hydrogen. The only liquid used to excite this battery consists of one part of sulphuric acid, and seven of water. The addition of a few drops of nitric acid makes it act with greater intensity, but it is not advisable to use it unless the silver is thoroughly covered with platinum.

Another form of this battery consists of a glass vessel like a tumbler, on which rests the frame which supports the metallic plates. As in the other, two screw caps on the top of the frame allow the attachment of wires for the conveyance of the current. One of these is connected with a central slip of platinum foil, on each side of which descend amalgamated zinc plates, connected above with the other screw. Like Daniell's batteries, a series of these may be connected together, by making communication between the alternate zinc and platinum plates.

Groves' Battery.—This is the most energetic battery known. Its activity is very great, and though this prevents it from being so well adapted for galvanoplastic operations, it is the one generally employed for the development of magnetism, and is in common use in the magnetic telegraph.

Various forms of this arrangement are met with, but in the most common one, a strip of platinum foil, furnished above with a screw cap, is immersed in a cylinder of porous earthenware, filled with strong and pure nitric acid. This cylinder is surrounded by another one of amalgamated zinc, also provided with a screw cap, standing on short legs, and divided by a longitudinal opening in one side, in order to permit the acid to circulate freely around it. It is placed in a glass jar or tumbler, containing one part by measure of sulphuric acid, and eight of water. When the circuit is completed by bringing together the wires placed in the screws, the hydrogen from the decomposed water in the outer vessel is not given off in the gaseous state, but passing through the diaphragm, combines with some of the oxygen of the nitric acid, reducing it. to nitric oxide. Some of this dissolves in the acid, and the rest escapes in the form of dense red fumes of nitrous acid, formed by its combination with the oxygen of the air.

This battery owes its intensity and rapidity of action to the absorption of the hydrogen, the good conducting nature of the materials, and the consequent concentration of the fluid. It has been said to be, when properly prepared, about seventeen times more powerful than that of Daniell. The great objection to its use arises from the escape of the irritating and poisonous nitrous acid, which is sometimes so considerable as to fill the apartment with the fumes.

Bunsen's Battery.—This is the same in principle as Groves' battery, but is more economical, as a cylinder of porous coal is used in place of platinum. It is represented in Fig. 389.



A B is a glass vessel filled up to B' B' with commercial nitric acid. C and C' are hollow charcoal cylinders, dipping into the acid as far as B" B", and resting on the edge of the glass by a flange. A ring of zinc or copper P encircles the top of the charcoal cylinder, and terminates in an appendage P', for connecting it with the wire. D D, which are diaphragms of porous earthenware, contain an amalgamated hollow zinc cylinder Z Z, with its appendage P", also intended for communication, and which is immersed in dilute sulphuric acid. The connections are made by means of the clamp A B, Fig.



390, and screw V, which are shown in place at H, Fig. 389. The perfect contact of these appendages, screws, and the ribbons or wires of copper connected with them, must be secured, by keeping them clean and bright by rubbing with sand paper. When the battery is about to be used, the glass vessel is half filled with equal parts of commercial nitric acid and water, and the

diaphragm, with water acidulated with sulphuric acid. The coal cylinder is prepared by pressing a thorough mixture of

# CONNECTION OF BATTERIES.

Starts Coal & & Ry flow are recordered by Veleree

one part of caking coal and two of coke with a little rye flour,<sup>+</sup> into a cylindrical mould of sheet iron, in the centre of which is a core of wood or pasteboard. The mould, after being closed by a movable cover well luted on, is heated gradually to redness, and the calcination is continued until the disengagement of gas ceases. The cylinder is then taken out, soaked in a strong solution of molasses, dried, and again calcined by an intense heat, in order to increase its firmness of texture. After this, its surface may be smoothed off with a file, or in a lathe.

This battery is said to be almost equal to Groves' in power. Professor Bird has constructed one similar to it by the use of a black lead crucible. He ignited the crucible for a short time, and, when thus prepared, filled it with nitric acid, and wound a wire tightly around its outside, making it serve both as a support and as the conductor of the fluid. A bar of amalgamated zinc, also connected with a wire, was then placed in a porous cylinder containing dilute sulphuric acid, and the whole was immersed in the acid of the crucible. He states, that, although powerful, it is much inferior to Groves' battery.

In the use of any of the above described batteries, care must be taken not to fill either of the receptacles too full of the liquid, since on immersing the metals or charcoal, some part of it might overflow and mix with that of the other vessel to the injury of the surfaces. After the insertion of the cylinders or bars, the surfaces of the two liquids must be as nearly as possible upon the same level; any deficiency in this respect being compensated by the addition of more fluid.

Connection of Batteries.-The connection between the dif-



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ferent plates of batteries is very conveniently made by means of the binding screw, Fig. 391. The wire by which the communication is established, is passed through the hole in the side, and kept in its place by the movable screw in the top. The screw below, serves to fasten the arrangement firmly into a hole of the proper size, in the top of either plate. The operator should be supplied with a number of these, as they permit him to unite and disconnect the different parts of an apparatus with the greatest ease and rapidity. They are shown in the figure, attached to the copper and zinc plates of a simple circuit, with the wires, of which the ends form the poles, passing through them.

Wire for Battery Purposes.—Copper wire is more often employed for connecting the different parts of a voltaic circuit than any other, on account of its high conducting power, its flexibility, and its not being susceptible of magnetization by the passage through or around it, of a galvanic current. Its thickness should be proportioned to the energy of the battery, and it should be as short as possible, because a great length of wire causes resistance to, and loss of the fluid proceeding from a battery of moderate power. Its connecting parts, as well as those of the plates or screws to which it is attached, must be bright and clean. In order to ensure perfect contact, it is advisable to amalgamate the extremities of the wire. This is readily done by washing them with a solution of nitrate of mercury, and dipping them afterwards in metallic mercury.

This coating is apt to oxidize, and thus to cause an interference with the complete connection. When this occurs, the film of oxide is to be rubbed off, and the amalgamated surface renewed as before. This may be done with perhaps greater ease than in the former method, by placing a few globules of mercury and a little tallow upon a piece of chamois leather, and then rubbing the wire with it until the mercury adheres to its surface. When the second coating is applied in this way, it is less apt to become tarnished than if made to adhere by the aid of the solution of the nitrate. When it is desired to break and renew the connections often or very rapidly, the common mode of attaching the wires is found to be inconvenient. In that case, a little cup made of copper, or other metal which does not too readily amalgamate with mercury, is partly filled with that metal, and the wires are received in the cup, a

### ELECTROLYSIS.

depression in the bottom of the latter being often made so as to hold them more firmly in their place. By keeping one of the wires immersed in this cup, the connection may be made complete or broken at will, and without disarranging any part of the apparatus, by simply placing the extremity of the other wire in its appropriate cup, or taking it out.

The wires are, in one point of view, the most interesting parts of the battery, as it is at their extremities or the *electrodes*, that the most important phenomena of galvanism are exhibited.

Electrolysis.—Any one of the batteries already mentioned. may be employed for the purpose of producing chemical decompositions, by passing the current from them through the substance, from pole to pole of the terminal wires of the series. As electro-chemical changes are usually effected most perfectly by a current of *intensity*, as distinguished from one of *quantity*, which is more active in producing light, heat, and electro-magnetism, a number of pairs of plates or cylinders, varying with the difficulty of the decomposition, are employed. The other results spoken of are generally obtained by using a small number of plates with large surfaces. A combination of small batteries, made upon the plan of Daniell's is, perhaps, the most active of all in producing chemical change.

Whatever form of apparatus is used for such decompositions, particular attention must be paid to the proper connection of the alternate metals, and to the close contact of the wires, as well as the other circumstances before spoken of in reference to their relative size. The points of the wires should in most cases be made of platinum, as that metal is the best conductor of the fluid, and is not liable to be chemically acted on by any of the substances evolved from the electrolyte.

Any one of the class of bodies called *electrolytes*, which includes all those known to be capable of decomposition by electricity, may be exposed to the voltaic influence by being placed between the *electrodes* or extremities of the wires, so as to be the medium of communication between them. This is effected in various ways, as the substances differ in being solid or fluid, and good or bad conductors of the influence.

Many solutions, like that of iodide of potassium, admit very readily of decomposition. A solution of this salt may be easily decomposed by a battery consisting only of a wire of zinc and one of copper. Water alone, however, may require 29 the power of a number of cells of Daniell's battery to separate it into its elements. The addition of a little common salt, or of almost any saline body, will make the electrolysis of it much more easy by increasing its conducting power.

In the decomposition of water, and indeed, in most cases in which gaseous components of bodies are eliminated from liquids, platinum strips are attached to the ends of the wires, thus making the surfaces of contact much greater. These strips, which may be made of platinum foil, are placed parallel, and as close to each other as is possible without their being actually in contact. Their touching each other would effectually prevent all chemical action, as the voltaic fluid would be directly transmitted through the wires from the positive to the negative plate.

When the electrodes are placed in a vessel of water, and the battery is made to act properly, bubbles of hydrogen will ascend from the end of the wire or foil connected with the negative end, and oxygen from that of the positive one. These gases can be collected in a tube closed at one end, or a jar previously filled with water, and inverted over the wires; or they may be separately received in different vessels. As water consists of two volumes of hydrogen and one of oxygen, of course the quantity of the first given off, will be twice that of the last. Fig. 392 represents a mode of effecting this de-



composition in which the terminal wires of a trough arrangement, are passed through a perforated cork into water contained in a funnel. The end of each wire is placed directly under a test tube previously filled with water, and inverted in the funnel. The ascending gases displace the water, occupy the tube, and may if necessary, be accurately measured.

As the quantity of electricity set in motion by the battery is in direct proportion to the amount of zinc dissolved in it, so are the effects of chemical decomposition always proportionate to the former; this being thus always in a certain relation with the equivalents both of the products of electrolysis, and of the portion of zinc acted upon. Thus, one grain of hydrogen, given off at the negative pole, indicates that thirtythree grains of zinc have been dissolved during the time of the action. Upon this principle Faraday constructed his voltameter, which affords the only means known of accurately measuring the galvanic influence. That form of this which is most employed, is one in which strips of platinum foil attached to the wires of a battery, are placed opposite and near to each other in a jar or bottle, from which a tube issuing, enters under a graduated jar inverted over the pneumatic trough, all of these vessels being full of water. By the measure of the gases collected, the quantity of electric force can be estimated. By placing slips of platinum upon the ends of the wires in Fig. 392, and substituting a single graduated tube with a wide or funnel-shaped mouth, for the two which are seen in the cut, the same result may be attained.

Faraday describes a convenient form of tube for the collection and examination of gases evolved from either electrode, in experiments conducted upon a small scale. This tube, represented in Fig. 393, is filled with the solution

to be acted upon, and held in the position represented. The nature of the gas to be collected, depends on the end of the battery which is fastened to the curved wire at a. The other electrode is to be loosely inserted at b, so as to allow the gas given off from it, to escape through the open orifice. It should not be placed so far within the extremity of the tube as to permit any bubbles of the gas to pass around the bend, and to mix with that in the upright limb. The wire b is to be removed when a sufficient

Fig. 393.



amount of gas has been collected, and the latter can then be transferred to a suitable vessel and examined.

The methods of subjecting substances to the action of the battery are very numerous. When the electrolyte is a fluid, it may be placed in any one of a great variety of suitable receptacles. In all cases it must be recollected that the electrodes should be brought as near together as possible, so that the small amount of the substance which is directly between them, shall have the full effect of the current concentrated upon it. Decompositions of a drop of fluid may be made by placing it upon a glass plate, and bringing the poles in contact with its sides. Larger quantities may be received in a watch-glass or other concave piece of glass, or in a cup of the proper size. A very convenient mode of subjecting liquids to the current, so that the results of the decomposition can be easily inspected by the observer, is that of closing one end of a piece of glass tube tightly with a cork, and supporting it in an upright position by passing one of the wires of the battery perpendicularly through the cork. The tube may then be filled with the liquid, and the other wire, bent downwards, may be immersed in it, and placed along side of its fellow. In nearly all such decompositions, the ends of the poles should be armed with strips of platinum foil, on account of the greater surfaces of contact presented by them.

When it is desired to direct the electrolytic influence upon a large surface of a liquid, a piece of platinum foil attached to one pole may be hollowed out into a cup-like form, and the substance may be placed in it; or the terminal wire may be made to support, and communicate with a platinum crucible, by being wound around it. The other wire can then be immersed in the liquid, and prevented from touching the vessel by the intervention of a piece of glass tube.

Production of Heat and Light by Galvanism.—The physical effects of galvanism, among which are the production of heat and light, result generally from the passage of a current of great quantity and of feeble intensity, through an insufficient or imperfect conductor, the resistance of the latter impeding the current, and increasing its calorific power. The batteries employed for fusion and deflagration generally consist of a very small number of pairs with extensive surfaces, which will develope a great quantity of electricity. Usually these are the best batteries for physical experiments, but occasionally those consisting of a large number of plates are found useful for such purposes. A single pair of very moderate size will effect these results in a small way. Thus, Dr. Wollaston fused a very fine wire of platinum by means of a

### HEAT AND LIGHT FROM GALVANISM.

small battery, made of a lady's thimble and a rod of zinc. We have before stated that the intensity or decomposing power of the galvanic fluid is increased by placing batteries in connection so as to multiply the number of plates. Batteries may also be associated together so as to increase their calorific and light producing power. Any number of troughs like Wollaston's may for this purpose be placed—not as before, end to end—but sidewise, and the cells at either end of each may be connected with the same cells of the others by two wires, going across the series, and so bent as to be in perfect contact with the last plates. The projecting ends of these wires on one side are to be used as the poles of the battery.

Daniell's, or any other of the cell batteries, can be made capable of producing the physical phenomena of electricity, by paying attention to the size and conducting power of the wires or other bodies to be heated; but the quantity of the fluid is much increased by connecting a number of them so as to make them equivalent to a single pair. This can be done by connecting together all the copper or platinum plates by means of wires, either soldered to them or inserted into the binding screws already spoken of. The zinc bars or cylinders are to be brought into contact in like manner, and the poles may be made by attaching wires to any two of the opposite pieces of metal.

The wires of such batteries should all be made of larger size than those which are employed in the ordinary arrangements.

When the electricity developed in a powerful battery is passed through conical pieces of charcoal placed upon its poles, and these are brought into contact, and then withdrawn to a short distance from each other, the interval becomes occupied with a brilliant spark or arch of flame, the light of which is often too vivid to be borne by the eyes. The heat given out is also very intense, and gases and other bodies are sometimes subjected to its influence for the purpose of being decomposed. Carburetted and sulphuretted hydrogen are both thus affected by it. The wires may be twisted around two pieces of fine, well-burnt charcoal, which are then brought together. The brilliancy of the spark or arch passing between the points of charcoal, serves often to indicate the power and good condition of the battery. When a very powerful current is set in motion, it is advisable not to make the contact by means of the hands, but to use insulated dischargers analogous to those employed in electrical experiments. The wires may be brought together and disconnected by means of clamps or small vices attached to wooden handles. These may be screwed on and taken off at pleasure. The charcoal used in these experiments must be of the best quality. It is properly prepared by packing pieces of box or other suitable wood, two inches long, and a quarter of an inch thick, in an earthenware crucible, and, after covering them up with dry sand, heating them until they cease to flame. The best pieces must be selected and preserved for use in a well-stoppered vessel.

Various substances ignite and burn with brilliancy between the galvanic poles. Metallic leaves or foil of different kinds may be conveniently burned by taking them up upon the point of one electrode, and bringing them in contact with a plate of polished tinned iron, which is attached to the other. In this way the different appearances and colors of their flames are shown.

A platinum wire stretched between the poles of a battery, will attain a red or white heat, and, if offering sufficient resistance to the passage of the fluid, may even be fused. It must not be too thin, as the electricity may be sometimes so much retarded as to produce no visible indications of heat. A wire of the proper size will often remain at a red heat for a great length of time if a constant battery is used.

The power possessed by the battery of igniting platinum wire, enables us to apply heat in situations in which it would be difficult or impossible to do it by other means. By its use, substances placed under water may be ignited or exploded, if necessary, at a great distance from the operator. Out of the laboratory, it may be employed for the purpose of exploding gunpowder in mines, or under ships, and in other positions far removed from the source of electricity; while, in it, it may be used for the explosive decomposition of various gases.

Dr. Hare has taken advantage of this power in the construction of his sliding-rod aqueous eudiometer. This instrument consists of a glass vessel, with a capillary orifice closed by a spring and lever in its top, and connected below with a socket, and a tube in which a graduated piston moves. A fine wire of platinum is stretched across the middle of the vessel, between two brass wires, which pass through the socket below, and terminate in legs, which are made capable of connection with the cups upon the poles of a battery. The instrument having been filled with water, the gaseous mixture is drawn into it in the proper quantities by pulling out the piston to regulated distances, and is then exploded by the ignition of the wire, after the capillary orifice has been closed. This last is now again opened, but under water, enough of which enters to supply the vacuum produced by the condensation. The amount of undecomposed air which remains, is indicated by the distance through which the rod has to be passed for the purpose of expelling it all from the glass vessel.

Dr. Hare uses for the ignition of the wire in this experiment, his calorimotor of two pairs of plates. He has constructed a variety of arrangements for procuring the heating effects of the battery. In one of these, twenty sheets of copper, and the same number of zinc plates, united separately to two bars of metal, were secured in a wooden frame, so as to leave a space between them of a quarter of an inch. A rope passing over a pulley, was attached at one end to the frame, and at the other to a counterpoising weight. The frame could be lowered by means of the rope into a cubical box containing the acid liquor. Another form of Hare's battery is so constructed that the vessel containing the acid is raised up to and lowered from the plates, when necessary, by means of a lever connected with pulleys. By this most convenient and powerful battery, constructed with a new arrangement of the plates, the most intense galvano-ignition and deflagration may be accomplished.

This apparatus, the description of which might, perhaps, have been more properly introduced along with the account of other batteries, is shown in Figs. 394 and 395. We extract the description of it from Hare's Compendium.

"The two forms of calorimotor represented by Figs. 394 and 395, have been much used by me for what is described in my Compendium as "galvano-ignition." (C, 335.) Within any cavity, ignition of any intensity short of fusing platina may be produced, by making a platina wire the subject of a galvanic discharge from an instrument of this kind. I first resorted to this process in the year 1820, for the purpose of igniting gaseous mixtures in eudiometers of various forms. In June, 1831, I applied it to ignite gunpowder in rock blasting; and to this object it was subsequently applied, agreeably to my recommendation, by Colonel Pasley, Professor O'Shognessy, and others.





"This machine consists of sixteen plates of zinc, and twenty plates of copper, each twelve inches by seven, arranged in four galvanic pairs. The plates are supported within a box with a central partition of wood, A B, dividing it into two compartments. Each of these may be considered as separated into two subdivisions, by four plates of copper between the letters C C. Of course the box may be considered as comprising four distinct spaces, No. 1, No. 2, No. 3, and No. 4. The circuit is established in the following manner. Between

the zinc plates of compartment No. 1, and the copper plates of compartment No. 2, a metallic communication is produced. by soldering their neighbouring corners to a common mass of solder, with which a groove in the wooden partition between them is filled. With similar masses of solder, two grooves severally made in the upper edges of each end of the box are supplied. To one of them, the corners of all the copper plates of space No. 1, and the zinc of space No. 4, are soldered. To the other, the zinc plates of space No. 2, and the copper plates of space No. 3, are soldered in like manner. Lastly, the zinc plates of No. 3 are connected by solder in a groove, and the copper plates of No. 4 are in like manner connected by solder in another groove. Upon the ends, SS, of the solder just mentioned, the gallows screws are severally soldered, and to these the rods, P P, called poles, are fastened. The means by which the acid is made to act upon the plates must be sufficiently evident from inspection. Depressing the handle causes the wheels to revolve, and thus, by means of the cord which works in their grooved circumferences, to lift the receptacle which holds the acid, until this occupies the interstices between the plates."

Means of Detecting the Galvanic Fluid.-The Galvanometer.-If a common magnetic needle, supported upon its pivot, be placed directly under and parallel to a wire which is connected with the poles of a galvanic circuit, so that the positive fluid will pass through the wire from the north to the south, it will, during the passage of the current, leave its position in the magnetic meridian, and, after a few oscillations, assume one nearly or quite at right angles to it, its northern end or austral pole pointing to the east, or to some point between it and the north. Precisely the same effect will be produced if the needle is placed over the wire, and if the direction of the current is reversed. But the northern end will be turned towards the west, if the current is passed from the north to the south while the wire is under it, and also in the same direction if the wire again placed over it, transmits the fluid from the south to the north. The needle always returns to its former position immediately after disconnecting the wire. The power possessed by a galvanic current of influencing the magnet, may be increased to almost any extent, by passing it through a number of wires, or a coil made of a single one, so as to make the action of the whole equivalent to the sum of the actions

of all its spires. This can be done most effectually by bending a long wire, covered with cotton or silk to prevent the lateral escape of the current, into the form of a rectangle. The needle is supported parallel to, and between its horizontal branches, and it is obvious that it will be similarly affected by each part of the coil, in whatever position its wires may be; for, as before stated, a current passing above it from the north to the south, and one passing below from the south to the north, cause it to deflect in the same direction. This instrument is the galvanometer, or the "*electro-magnetic multiplier*" of Schweigger. By its use we can detect traces of electricity much too minute to act on the gold-leaf electrometer; but its chief applications are to the discovery of delicate galvanic currents, and to the determination of their direction. As



shown in the figure, it consists of the coil of covered copper wire NBS, containing usually about twenty convolutions, of which the extremities are connected with the cups CZ. A card graduated into 360° is fixed to the board A, so that a line drawn between the numbers 360 and 180, coincides with the direction of the centre of the coil. Above this is placed a delicate magnetic needle, supported on a pivot. The coil is placed with its long axis in the magnetic meredian. If any source of feeble electricity is now connected with the cups, the current from it will pass through the coil, and the magnet will move to the east or west, according to the direction of the fluid. The intensity of the influence is estimated in degrees, by comparing the position of the utmost divergence of the needle with the number under it on the card. The delicacy of this instrument depends in a great measure upon the number of convolutions of wire. Thus, if all other circumstances are favourable, it may be supposed that one consisting of one hundred turns will detect an amount of electricity which is only one-fifth as great as that shown by the one with twenty convolutions.
#### THE ASTATIC GALVANOMETER.

The Astatic Galvanometer.-The sensibility of the common galvanoscope may be almost indefinitely increased by connecting the magnetic needle immovably with another one placed above the rectangular coil of wire, but parallel, and opposed in the direction of its poles to the first. They are fastened by their centres to a common axis, which revolves freely in an aperture of the upper branch of the coil. This axis is suspended by a fibre of silk to the upper part of the glass or other vessel in which the whole is encased, and it penetrates a graduated card, placed under the upper needle. This arrangement makes the needle a balance of torsion, the movements of which are compared with the degrees marked upon the card, in the same way as in the simple multiplier. Terrestrial magnetism has scarcely any effect upon this system of needles, and would have none at all if both possessed an equal amount of magnetic power, the tendency of the one to assume its position in the meridian being in that case entirely counteracted by the reversed direction of the other. By a reference to the statements at the head of this article, it will be seen that a current passed through the coil, in either direction, will have the same effect upon both needles. Fig. 397

Fig. 397.



represents two of the many forms of Nobili's galvanometer. It is called *astatic* because it is unaffected, or nearly so, by the magnetism of the earth.

As these instruments are used not only to detect currents, but also to ascertain the directions in which they pass through the wires, it is of importance to impress upon the mind the movements of the needles which indicate that one or other extremities of the coil are in connection with the positive or negative electric poles. A simple aid to the memory is to suppose that a current is passing around the middle of a watch, from the handle over the face, and is returning back to its place of origin. The minute hand, if pointing to the hour twelve, which is usually placed next to the handle, may be supposed to represent the northern half of the needle. It would then move around in its usual direction towards the figure three. If the current were passed around the back of the watch from the handle, and returned to the face, the hand would move backwards towards the figure nine. Ampere has devised the following formula, which is still better calculated to impress the direction of the deviations upon the memory. "Let any one identify himself with the current, or let him suppose himself to be lying in the direction of the positive current, his head representing the copper, and his feet the zinc plate, and looking at the needle, its north pole will always move towards the right hand." The person must, however, suppose himself to be lying over the needle, his head and its north pole being both in the same direction.

Our limits would not permit us to refer to the applications of galvanism to electro-magnetic apparatus, or the electrotype, even if these were more pertinent than they are to our subject. A full account of them is to be found in a number of popular treatises. "Davis' Manual of Magnetism," and Walker's "Electrotype Manipulations," contain a full description of all the means employed in experiments on these subjects, and of their practical applications.

# CHAPTER XXXI.

#### CONSTRUCTION OF FORMULÆ.

ALL compounds are either mechanical mixtures following no precise law, or consist of simpler bodies united in definite proportions agreeably to the laws of chemical attraction. The latter may be represented by formulæ. There are many advantages attending the employment of formulæ, and nothing has tended to advance the science of chemistry further and more rapidly than their use. They convey to the eye, like pictures, a far clearer view of the nature of a compound than the most labored description could effect. While they are established by analysis, their reaction tends to confirm or disprove its results. As they are pictorial representations, the memory may retain the composition of thousands of compounds, and yet not be overburdened. Isomorphous bases may be thrown together under a short and general expression, and thus substances, often differing widely in external properties, are brought into natural groups, a result to which the analysis of a body would never lead without the formula.

When a definite compound has been separated by analysis into its constituent parts, their relative proportion is generally expressed in per centages, but such a mode of expression does not convey a clear idea of the chemical nature of the body, as compared with other compounds, containing the same or allied constituents. The per centage composition is usually given as simply expressing the results of analysis. To ascertain the nature of the union among the constituents, agreeably to the received laws of affinity, they must be reduced from their per centage proportion to the proportions of their equivalents. If any one of the constituents happens to express in the per centage results, the combining weight of that body, the others will also express their combining weights or multiples of them. Or if any one can be multiplied or divided by any number, which will give the combining weight of that body, the others multiplied or divided by the same number, (in order to keep up the same proportion as in the per centage results,) will express their combining weight or multiples of them.

Thus the analysis of carbonate of lime, according to Dumas (1), and Erdman and Marchand (2), gives-

	1	2	-2
Lime	56.06	56	28
Carbonic acid	43.94	44	22
	100.00	100	50

If the 44 carbonic acid be divided by 2, it gives the combining weight of one equiv. of the acid; and the lime if divided also by 2, gives the combining weight of one equiv. of it. It is, therefore, composed of 1 equiv. of each constituent. Again, if 56 be divided by the combining weight of lime, 28, the result is 2; and 44 divided by the combining weight of the acid, likewise gives 2. The proportion between the equivs. is, therefore, 2:2, or reduced to the lowest term 1:1.

Now since the per centage composition expresses the proportion between the combining weights of the constituents, if each constituent be divided by its combining weight, the result will be the proportion between the number of equivalents in the compound.

Then the lowest of these numbers divided by itself gives unity; and the others divided by the same number will express the proportion between all the equivalents, and generally in whole numbers, if the analysis has been correct. Thus the analysis of blue vitriol, by Berzclius, gives the following numbers in the 1st column, expressed in 100 parts.

Oxide of copper	32.13	0.803	1.018	1
Sulphuric acid	31.57	0.789	1.000	1
Water	36.30	4.0333	5.111	5

The constituents being severally divided by their combining weights, the numbers in the 2d column result; and by dividing each of these by 0.789, we get the 3d column, which, by making a slight allowance for the imperfections of analysis, gives the proportions between the equivalents 1:1:5.

Having determined the number of equivalents, a formula is easily established, which in the case of carbonate of lime is  $CaO, CO_2$ , and of blue vitriol  $CuO, SO_3 + 5HO$ . Now the per centage composition of dry or anhydrous sulphate of copper is 50 oxide of copper and 50 sulphuric acid. If we compare it with the per centage composition of blue vitriol, the relation between them is not readily seen; and in the case of many other substances, no relation whatever can be detected; but if the formulæ deduced from each analysis be compared, their relation is at once evident, for we perceive that the blue vitriol contains 5 equivalents of water, which the other does not, and that otherwise they are one and the same substance.

The silicates form a numerous class of crystallized minerals, whose formula may be established by the foregoing method, or by determining the quantity of oxygen in each element, and bringing these quantities into whole numbers, those of the isomorphic bases being added together. It is, perhaps, a

# CONSTRUCTION OF FORMULÆ.—GLASS-BLOWING. 455

more convenient method for these bodies than the preceding.

The construction of the formula for an organic body depends on precisely the same principles, and is ascertained by a similar process; but there is a peculiarity in the formula of organic bodies, which is rarely met with in mineral substances. Thus the analysis of olefiant gas gives as its formula CH; but from its density and other circumstances it should be C.H. or  $C_4H_4$ . The formula deduced from the analysis of sugar is CHO, but it decomposes into alcohol and carbonic acid, and, therefore, either  $C_{2}H_{3}O + CO_{2} = C_{3}H_{3}O_{3}$  ought to be the formula, or C<sub>6</sub>H<sub>6</sub>O<sub>6</sub> or C<sub>12</sub>H<sub>12</sub>O<sub>12</sub>, which last, indeed, is most generally received. A formula may also be doubled, or trebled, if viewed as a bibasic or tribasic acid. Therefore, after determining the simplest formula for an organic body, its more rational formula is determined by the specific gravity of its vapor, by its mode of combining with bases or acids, or by its metamorphoses.

# CHAPTER XXXII.

#### GLASS-BLOWING.

THE ability to work glass over the lamp or blowpipe-flame is a very desirable accomplishment for the chemist, as it enables him to fashion for himself, and in accordance with his own judgment, such micro-apparatus as is constantly in demand during experimental research. The inconvenience and expense of having a large stock of delicate glass instruments always at hand, and the difficulty of obtaining such at all times, especially in localities distant from the cities, render instruction in the art doubly desirable. On these accounts, we think it proper to devote a chapter to a few illustrations of the processes by which tubes are bent, closed, rounded, widened, and drawn out, and by which bulbs are blown and joints sealed.

The two principal pieces of apparatus required are a lamp and a table blowpipe. The latter, as well as its management, have already been written of at page 59. The former, known

# 456 DANGER'S GLASS-BLOWER'S IMPROVED LAMP.

as Danger's "glass-blower's improved lamp," of form shown by Fig. 398, is of sheet brass, and rests upon a tray designed



for the reception of any overflow of oil. These lamps are fitted with an arrangement by which the wick may be raised or lowered, and the flame consequently enlarged or diminished as desired: an accompanying hood, serves to increase the heat and to protect the eyes from the smoke and flame.

The wick may be made of common candle wick, divided into lengths of proper dimensions, and stranded together, so as to form a diameter of about three-fourths of an inch. This bunch is placed in that part of the lamp intended as its receptacle, and should only protrude above the oil about the third of an inch. When it is desired to lessen the power of its flame, as may be necessary in the heating of small tubes, the force of the blast can be diminished so as to make the flame of the desired height and intensity.

The fuel may be olive, lard, sperm, or tallow oil;—the latter, however, being preferable on account of its giving a hotter flame. In case of the absence of a lamp of this sort, any common metallic vessel, of proper size, may be fitted for use, upon the blowpipe table, by training up upon, and allowing to overhang its side, a thick bunch of wick. This may be kept in place, and its flame, at the same time, be prevented from descending too far, by encircling it with a tin or other metallic tube, or a coil of wire, which may be temporarily connected with the sides of the vessel, so as to answer all the intentions of support and the conduction off of the excess of heat.

If the experimenter cannot have access to a properly made blowpipe table, he may, in a very short time, construct a substitute himself, which, however rough, will enable him to carry on nearly all the operations of glass-blowing. A hollow reed or piece of cane angle, about a foot in length, may be firmly fixed in a circular hole, drilled near the edge of a common table, and which is just large enough to admit and hold it firmly in its place. This may have adapted, by means of cement, plaster or putty, to its upper end, a nozzle of metal, or of glass drawn out to the proper sized orifice, or one made of a piece of tobacco pipe of the requisite calibre. A bladder of the largest size, or bag of caoutchouc, furnished with two openings upon the same part of its circumference, is now firmly attached to the bottom of this tube, by one of these a similar piece of reed, long enough, however, to reach from the operator's knees-while sitting-to his mouth, having been inserted and tied into the other opening. That end, of this last-mentioned tube, which is within the bladder, should be provided with a valve, like that of a cupping glass, made by placing, loosely over it, a long strip of oiled silk of the diameter of the tube, folding the ends upon the body of the reed and tying them firmly to it by waxed thread. This valve admits of the passage of air into the receptacle, but will not allow its return through the same orifice, so that pressure upon the bladder will compel its exit through the nozzle of the tube which is fixed in the table. If now the operator, sitting near the table with the bladder hanging between his knees and the loose tube fixed in his mouth, inflates the former, and then presses upon it uniformly with his knees, a continuous current is expelled from the nozzle upon the flame of the wick placed directly above it. A repetition of the inflation only becomes necessary when the bladder is nearly emptied of its contained air. The inflation of this homemade apparatus is scarcely, if at all, fatiguing, and it permits to the glass-blower the unincumbered use of both his hands.

The position of the jet upon the top of the table, and that of the operator before it, are shown in the annexed drawing.

When it is desired to use the gas flame, which is, however, not so good as that of oil, the straight jet and Argand burner, as is shown in the above drawing, are employed. It is still better, in ordering a blast table, to have prepared a jet, with ball and socket joint suitable to either kind of flame, and

Fig. 399.



which can be screwed to, or removed from, the table at pleasure.

The other implements are an iron piercer with wooden handle, Fig. 400, a cone of biscuit-ware, Fig. 401, for widen-



ing the necks of tubes, a small pair of brass tongs, Fig. 402, for fashioning bulbs, &c., a small piece of smooth hoop-iron,

styled the *marver*, a hardened cast-steel knife, and one or two three-cornered files for cutting tubes and rods.

In addition to the above, the table should be supplied with a stock of tubes and rods, of assorted diameters, and made of glass free from lead. They should, moreover, be very uniformly regular throughout, and exempt from flaws or striæ.

Before commencing operations, the wick must be evenly trimmed and parted in the middle, so that when the jet is placed opposite in the rear, and in proper relation, it may drive the flame forcibly in advance, but not of too great length, else it will become smoky.

The tubes or rods, previously divided\* into the required length, should always be wiped perfectly dry before being subjected to the action of the flame, and then carefully and gradually heated, the uniform diffusion of the heat being effected by keeping them revolving;—these precautions, which are always to be observed, prevent breaking from sudden and unequal heating. After being heated, they must be removed gradually from the fire, and laid upon a piece of charcoal, so as to become annealed, as it were, by gradual cooling.

The most simple and easily performed of all the operations



of glass-blowing, is the rounding of edges, which is readily done by heating them to softness in the flame during constant

\* Tubes can very readily be severed, or divided into lengths, by scratching them with a file, and breaking asunder as at Fig. 407. For large tubes, the scratch must extend entirely around the circumference.

Vessels of larger diameters, such as necks of retorts, and the like, require the use of a diamond spark. According to Mr. Nasmyth, coke has the property of *cutting* glass, and can very well be substituted for the diamond.

When the scratch of the file is insufficient to effect a smooth division, moisten the scratch, and trace it with a heated wire or pastile. A heated wire will also divert a crack in a glass vessel to any desired direction. revolution of the tube between the thumb and three fingers, which support it. This operation, by which the edges of tubes and rods are smoothed, is also preliminary to that of widening the mouth of a tube, a test-tube for example, which is done by spreading it while hot, as shown in Fig. 403, by means of the iron piercer, or, better, the biscuit cone, either of them being previously warmed, and then carried round the opening with an outward pressure.

Tubes Cemented.—Tubes or rods are also cemented together by softening their ends and blowing gently through them at



the moment of junction. Care must be taken to hold them firmly and perfectly even, as shown in Fig. 404, and to retain hold of the joined tube until it has entirely cooled, else it

may bend by its own weight at the heated part, and thus become crooked.

If the tubes to be cemented are of unequal diameters, the



wider one must be *drawn out* at its end, so as to reduce it to the size of the smaller, and then be joined to it as above directed, and shown in Fig. 405.

Rods are cemented together by partially fusing their ends and bringing them carefully together, and pressing them until they adhere. The welding is then completed by heating the new joint, during which process, in order to impart shape, the rods must be kept rotating, and be alternately drawn out and brought together, until the junction is as smooth and uniform as any other part of the surface.

Tubes Bent.—Very small tubes can be bent over the spirit lamp, Fig. 115; but larger ones require the force of the blowpipe-flame to heat them. The operation of bending consists in heating the tube, to dull redness, about an inch on either side beyond the point of the intended curve, and just at the commencement of softening, in making an angle, by bending it dexterously but slowly in the desired direction until it assumes the required form. In order to prevent a flat, wrinkled, and consequently very fragile elbow, it is necessary to close the tube at one end, and blow gently into the other, during flexion, so that the pressure of the air within may counteract any tendency to malformation.

Drawing Out.—When a tube is to be drawn out, either as preliminary to further working, or in the preparations of nozzles for washing bottles, or other purposes, one of the proper size is taken, at the ends, between the thumb and index of each hand, and along its length with the other fingers, and kept revolving gradually over the flame until it becomes red, and commences to soften at the heated part. It is then taken from the fire and drawn apart, as shown in Fig. 406.

# Fig. 406.

In this way also stirring rods are pointed, and when the tips of either tubes or rods thus wrought are to be smoothed, it is only necessary to divide, or break across the centre of the part drawn out, and to heat the surfaces in the flame until

they soften and fuse. The proper mode of severing glass rods or tubes, is first to make a deep scratch with a three-cornered file in the spot where separation is required, and then, after grasping

them as shown in Fig. 407, by gently breaking them apart.

The tube must not be kept in the fire too long, nor yet drawn out too rapidly. When the tube or rod is too short to be divided, it may be drawn out at either of its ends by means of a *punto*—a piece of glass rod which is heated to softness and cemented to the other as a handle.

Tubes Closed.—Very small tubes may readily be closed by softening their edges over a flame, and rotating them until they unite and adhere. Tubes of larger size are treated in the same way, but to facilitate their closure, occasional pressure of the hot end against the back of the tool, Fig. 402, and sometimes gentle blowing through the open end, are required. Tubes also are closed hermetically by drawing out one end, as shown Fig. 408.

in Fig. 408, by then scratching with a file and breaking asunder the part a, and finally by closing the small orifice by fusion in the flame.





#### DRAWING OUT AND CLOSING.

Drawing Out and Closing.—When it is desired to form a vessel like a test-tube, a tube of the required diameter is drawn out, as at Fig. 409, and then cut as under at a. The



two pieces thus formed, serve to make two test tubes. For that purpose, it is necessary to heat the smaller end of each to softness, and immediately upon removal from the flame, to blow cautiously and slowly into the open extremity until the closed end assumes a uniform spherical shape. Sometimes it

Fig. 411.

is necessary to repeat the heating and blowing in order to fashion the bottom perfectly, as seen in Fig. 411. If the piece of glass is only long enough to form one tube, its end can be drawn out by attaching a punto, as before described, and now shown at b, Fig. 410. This punty, or glass rod

handle, serves also to remove any redundant glass, it being only necessary for that purpose to heat the closed end highly, to apply the punty a little less heated, and after collecting upon its end as much of the surplus melted glass as is required to make the bottom thin and capable of supporting sudden changes of temperature, to draw it off. This manipulation requires some dexterity, which is, however, easily acquired by slight practice. If at one heating and gathering the bottom has been reduced to the proper thinness, it may be heated anew, removed from the fire, and then by slow and gentle blowing, through the open end, the bottom may be blown out to roundness. The mouth of the tube is then finished as directed at page 460, Fig. 403.

Lateral Attachments.—To attach a tube to the side of another is somewhat difficult. For this purpose, the tube with which the junction is to be effected is closed at one end and heated at the desired point, such as b, Fig. 412, to high redness. To this hot part a glass rod, or punto c, slightly heated, is attached and drawn out, as shown in the figure. When the glass has cooled, cut off the new joint at



b, heat again in the flame, and widen its mouth with the tool, Fig. 400, to the size of the diameter of the tube which is to be joined with it. This having been done, the tube is to be attached as directed at page 460. Fig. 413 shows the joint perfected.

Another mode is to heat and close the drawn out end, and to blow forcibly through the tube until the bulb, thus formed, bursts. All the remains of the thin glass bulb being broken off, a protruding aperture is left, to which the lateral tube may be cemented, in the usual way, by heating the edges of the ends of the two tubes to be united, joining them in the flame with slight compression, heating the joint to redness, and then slightly blowing, to give form and prevent cracking.

To Blow Bulbs.—To form a bulb at the end of a narrow tube, it is only necessary to continue heating it after closure until it commences to soften, and then immediately upon its removal

from the flame, to blow into the open end, as in Fig. 414, slowly, until the heated part expands to the proper size and shape. Care must be taken to heat the tube to a sufficient extent, so that there may be enough glass softened to give a bulb of the required size;—moreover, during both the heating and blowing the tube must be kept slowly rotating between the fingers, so as to prevent an accumulation of the melted glass, by its own weight, in any one part.

Fig. 414.



To blow a bulb in the middle of a tube, the latter must be heated at its centre during constant but slow rotation between the fingers, and then carefully blown into at one end, whilst the other is closed with the finger, a cork, or a piece of wax. The pressure of the air within expands the hot glass into a spheroid, regular or irregular in form, according to the care and skill of the operator. The part of the tube to be expanded must be heated uniformly and kept in constant and slow revolution during both the heating and blowing.

In the fashioning of certain glass-tube apparatus, it is sometimes necessary to blow the bulbs separately, and to attach them afterwards to their adjacent parts;—the bulb is then formed as follows. Take a glass tube A, Fig. 415, of



the required diameter and length, heat it at the points a and b, and draw it out in two places, as shown at r s in B. When the tube has cooled, divide it at the attenuated parts r s with the file, as directed at page 459, Fig. 407, and close one end of one of the pieces in the flame. Then hold it by the other end, which is drawn out, heat it to redness, and fashion the bulb by blowing, as above directed, until it assumes the shape of G. It is then cemented to the other parts of the apparatus, as directed at page 460, the previous widening of the drawn out parts being performed as at Fig. 403.

Thermometer bulbs are made by expanding, as above directed, the heated end of tubes with a capillary bore.

To Make a Welter's Tube.—By way of illustrating the different operations of fashioning glass tubes over the blowpipe-flame, we will go through the different stages of manufacture of the safety tubes of Welter. A straight tube is first bent into form, as at A, Fig. 416, and the flame is directed upon *a*; as soon as the glass softens at that point one end of the tube is closed with the finger, and the other is blown into

# GLASS-BLOWING: WELTER'S AND FUNNEL TUBES. 465

forcibly, so as to form the very thin, brittle bulb represented Fig. 416.



by the dotted lines. When the tube is thick a repetition of the heating and blowing is required. This bulb is then

broken off, and the bent tube, thus formed, is ready to be attached to the straight tube B. This latter is formed of a separate tube, and having a bulb bblown into its centre, is cemented to A at a, in the manner before directed. The funnel top of the tube B, is formed by first blowing a bulb c on its upper end to extreme thinness, removing it with the file and cementing a bulb, with open mouth, as at x in D. The S form is given merely by bending B in the proper direction.

Instead of an open *bulb* at the top of the D tube, a small funnel is cemented to it, as in the fashioning of funnel tubes, Fig. 417. Fig. 417.

# CHAPTER XXXIII.

#### CORKS.

CORKS are in many ways indispensable for laboratory purposes, and the stock should consist of all sizes; those for mounting apparatus being necessarily of the finest velvet kind, smooth and as free as possible from imperfections.

An excellent means of increasing the elasticity of corks is compression by a small apparatus, Fig. 418, sold for the pur-

Fig. 418.

pose. This treatment renders them capable of being fitted to apertures with great nicety and ease.

We have frequently made mention of the adaptation of tubes and other parts of apparatus by means of perforated corks. These perforations may be made with a hot metallic rod and afterwards enlarged with a rat-tail file; but a much smoother and neater hole can be made with a cork borer, Fig.



419, which is intended specially for this purpose. It consists

of a series of brass tubes of uniform length, but varying from an eighth to one inch in diame-

ter, and fitting one within the other. The sizes contained in such a series are equal to all the requirements of the laboratory, as holes of smaller or larger dimensions than the above extremes are seldom required. Each of the tubes is open below, but closed at the top with a cap c, Fig. 420, through which is a hole b for the passage of a stiff wire d, which serves both as a handle and as a punch for ejecting the cores from the tube after the perforation of the cork.

The drawing exhibits one of the tubes of the series already in operation, it being only necessary to bring its base upon a cork, and to effect

the perforation by pressure upon the cap and a slight circular motion. The core or part of the cork removed, ascends into the barrel a of the tube and must be ejected by the force of the punch d. As the tubes become dull on the edges, they may be sharpened upon a grindstone or with a fine file.

As a familiar illustration, we exhibit in the Fig. 421 a cork



thus treated with tubes inserted in the perforations. Their convenience is shown in many of the arrangements of which we have given drawings.

When the corks are not of good quality, they may be rendered impermeable by coating their surfaces with soft cement.

India rubber corks have lately appeared in the market;they are made by Goodyear, and answer admirably as cheap stoppers of bottles containing substances which are volatile, and which do not corrode the caoutchouc.



# CHAPTER XXXIV.

## DEALERS IN AND MANUFACTURERS OF APPARATUS.

For the convenience of those engaged in the study, practice, or teaching of chemistry, we here introduce the address of a number of prominent dealers in and manufacturers of the required furniture and reagents. From one or more of them may be obtained each and every implement and article mentioned in this work. Some few issue catalogues of their articles, with the price of each affixed, and furnish them gratuitously upon application;—their names are designated in the list below by asterisks.

Joaquim Bishop, Laurel Street, Philadelphia; manufacturer of platinum vessels, and of all kinds of chemical or philosophical metallic apparatus.

Bently & Co., Baltimore; manufacturers of portable steam generators; Morris, Tasker & Co., Agents, Philadelphia.

Charles Button\*, 146 Holborn Bars, London; dealer in chemical apparatus, and manufacturer of pure chemicals.

J. P. Duffey, South Eighth Street, Philadelphia; manufacturer of delicate balances, and of chemical and philosophical apparatus generally.

Wm. Debeuist, University of Pennsylvania, Philadelphia; manufacturer of metallic apparatus.

Joseph Fisher, 58 Chestnut Street, Philadelphia; manufacturer of thermometers and hydrometers.

L. C. Francis, No. 13 Dock Street, Philadelphia; manufacturer of thermometers, barometers, and of metallic apparatus.

James Green, Baltimore; manufacturer of electrical and other metallic apparatus.

Richard Griffin & Co.\*, Glasgow, Scotland; dealers in chemical apparatus and reagents.

J. G. Greiner\*, Berlin, Prussia, manufacturer of accurate thermometers and hydrometers for experimental research.

# DEALERS IN AND MANUFACTURERS OF APPARATUS. 469

B. B. Gumpert, 120 North Second Street; manufacturer of electro-magnetic machines.

Hammet and Hiles, 128 Vine Street, Philadelphia; manufacturers of copper hollow ware.

Haig & Co., 545 North Second Street, Philadelphia; manufacturers of blue stoneware.

Stephen Heintz, Queen above Warren, Kensington, Philadelphia; glass blower and manufacturer of all kinds of tube apparatus.

Hartell and Lancaster, Union Glass Works, Kensington, Philadelphia; manufacturers of tube apparatus, and of all other kinds of chemical glassware.

Hansell, Pine, above Tenth Street, Philadelphia; turner in wood, and manufacturer of supports, clamps, and filter stands.

Edward N. Kent\*, 116 John Street, New York; general depot for the sale of ALL kinds of chemical and philosophical apparatus, and of pure chemicals.

Lindsay & Blakiston\*, North-west corner of Fourth and Chestnut Streets, Philadelphia; publishers and importers of scientific books.

Abraham Miller, Zane Street, Philadelphia; manufacturer of assay furnaces and of all kinds of pottery.

Alva Mason, South Fifth Stréet, Philadelphia; manufacturer of chemical and philosophical apparatus.

Powers & Weightman, corner of Ninth and Parrish Streets, Philadelphia; manufacturers of acids and pure chemicals, and of chemical glassware.

Z. Pike, New York; manufacturer of chemical and electrical apparatus.

Mauldin Perrine, Baltimore; manufacturer of blue stoneware retorts, adapters, crystallizers, digesters and other apparatus, of the same material, for chemical uses.

Bullock & Crenshaw\*, North-east corner of Sixth and Arch Streets, Philadelphia; manufacturers of and dealers in pure chemicals, glass and porcelain apparatus.

Savery & Co., corner of Reed and Front Streets, Philadelphia; manufacturers of plain and enamelled hollow-ware of iron.

Tatham & Brothers, Philadelphia; manufacturers of smooth lead-pipe.

L. Voigt, North Third, above Vine Street, Philadelphia; Agents for Storms and Fox's glassware.

#### 470 DEALERS IN AND MANUFACTURERS OF APPARATUS.

Jas. M. Wightman\*, 33 Cornhill, corner of Franklin Avenue, Boston; manufacturer of metallic chemical and philosophical apparatus.

E. Wight, No. 4 South Fifth Street, Philadelphia; manufacturer of philosophical apparatus.

Weiss & Schively, 43 North Front Street, Philadelphia; importers of Beindorff's portable laboratories; of glass and porcelain ware, and of fine drugs and chemicals.

Samuel Wenzell, corner of Queen and Palmer Streets, Kensington, Philadelphia; manufacturer of laboratory tables, mineral cases, and wooden apparatus.

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