A handbook of chemical manipulation

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Section XXV. Electrical and Galvanic Manipulation

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SECTION XXV.

ELECTRICAL AND GALVANIC MANIPULATION.

654. The manipulation connected with the above subjects having been so elaborately treated of in numerous works specially appropriated to them, it becomes unnecessary to enter upon them here, except so far as they bear directly upon chemical research, and therefore those operations only will be alluded to which are required in laboratory work.

The investigation of Kolbe on the electrolysis of organic bodies, has opened a new field of investigation which is likely to be followed up with activity; the researches of M. Becquerel upon the separation of bodies by electrolysis, and even the application of the electrotype to the coating of chemical vessels (§ 683, p. 430), render it necessary for the student not only to possess the knowledge derivable from works exclusively treating of electricity, but also to be familiar with the means adopted for applying the electrical forces in chemical processes.

655. The operations to be detailed are all of the simplest character, but none the less necessary and useful.

656. The methods of exploding mixtures of gases in the various kinds of eudiometer have already been shown (§§ 551 to 558), and it was mentioned that in some cases a spark from the prime conductor of an electrical machine was insufficient to cause explosion, and the means of overcoming this difficulty were pointed out.

657. It sometimes happens in laboratories that it is desired to make eudiometric experiments, and that an electrical machine is not at hand. In this case the electrophorus will answer equally well, and from the ease with which it is constructed, and, if necessary, repaired, the small space that it occupies, and its freedom from the liability to fracture possessed by cylinder and plate machines, it is even more convenient. To construct it, a plate of tin is made into a circle of about 12 inches diameter, a raised border is then turned up for about $\frac{1}{2}$ an inch, and the ex-

treme edge is turned outwards over a wire, so as to avoid a sharp border. A mixture of equal weights of shell-lac, Venice turpentine and resin is made by gently heating them together until well fused, stirring during the time with a stick, so as to thoroughly incorporate the ingredients. The composition should, when perfectly homogeneous, be poured into the plate with the raised edges until it is quite full, and the composition is to be kept melted (but not too hot) for a short time, or until the bubbles have entirely disappeared. The portion which serves the place of a prime conductor consists of a flat circle of wood, rounded on the edges, and neatly covered with tin-foil; it is rather less in diameter than the lower portion of the instrument. A handle is easily constructed out of a piece of glass rod, and is fastened with electrical cement into the centre of the wooden plate. A brass ball may be attached to the rim; it is, however, scarcely necessary.

658. The tin plate with its resinous surface being warm and free from moisture, is to be excited by beating with a piece of warm flannel. To do this effectually, the latter is to be folded into a slip about 14 inches long, and being grasped by one end, is to be struck upon the resinous surface smartly and in an oblique direction, much in the manner of beating dust off a flat surface. When this has been done for about a minute, the warm dry cover or upper plate of the instrument is to be placed upon the resinous cake and touched with the finger. If the plate is then raised a few inches and the knuckle approached, a powerful spark will pass; and if the plate be again replaced, touched, &c., and so on in the same order, the knob of a Leyden jar being substituted for the finger when touching the upper plate after raising it, a jar may be charged sufficiently to give a powerful shock, and amply sufficient to cause the explosion of a gaseous mixture even under the unfavourable circumstances previously described.

659. The ordinary cylinder and plate electrical machines are so familiar to all persons in any way conversant with philosophical apparatus, that it will be unnecessary to describe them here, and it is almost equally unnecessary to remind the operator that

before use the instrument should be dry and even slightly warm; care must, however, be taken that the machine is not placed sufficiently near the fire to injure it, or fuse the cement by which the cylinder is fastened into its bearings, or the pillars into their sockets. The dryness of the glass rod which supports the prime conductor must also be especially attended to.

660. Frictional electricity is but little used in chemical experiments, while, on the other hand, the fluid, in the state in which it is obtained by chemical action, or, in other words, by the use of galvanic batteries, is in frequent requisition in the laboratory. The extreme purity of the gas evolved by the action of the battery on water renders it peculiarly adapted for eudiometrical experiments.

661. Many different forms of battery have been contrived, all being perhaps more or less valuable under certain circumstances; there are, however, some which are especially so, either for convenience, power, or economy. With regard to the former of these qualifications, Smee's battery is beyond all doubt preeminent. It is, it is true, less powerful than some other arrangements, and in constancy is decidedly inferior to the arrangement of Daniell; but the ease with which it can be constructed and repaired, the fact of its only requiring one exciting liquid and no porous cells, and especially not creating any unpleasant or corrosive vapour, make it so much superior for general laboratory use that its construction will be somewhat minutely described.

662. *Smee's battery.*—Each cell contains three plates, two of active and one of inactive metal. The two active plates are of zinc, and the inactive of platinized silver.

Zinc being almost invariably used for the active element in voltaic arrangements, it will be proper to describe a few precautions affecting its application in all cases. Ordinary zinc is susceptible of both common chemical and also of a local action, which takes place upon merely dipping the metal into the dilute acid used to excite the battery. The local action arises from the presence of impurities, which cannot be removed without causing the metal to lose one of its chief advantages, namely, its cheapness. But, by amalgamating it, the actions alluded to are prevented, and the crude metal works equally well with the pure. To amalgamate zinc, it is merely necessary to place the plates in a dish containing dilute sulphuric acid, and rub them with a rag, at the same time allowing a few globules of mercury to fall on them. The rubbing will cause the mercury to spread, and it may in this manner be made to entirely cover the whole surface of the plates. The latter should then be rinsed with clear water and put aside in a vertical position to allow the excess of mercury to drain off. By this means the surfaces of the plates acquire, after standing, a beautiful frosted appearance, and local action is prevented. Zinc plates which have been made from rolled metal are far better than when cast, and generally much purer; being smooth, they also work more pleasantly. The plates may be of any convenient thickness, but about $\frac{1}{8}$th of an inch is perhaps the most convenient. They are easily divided, by first greasing them, and then making a deep scratch with any convenient tool in the direction in which the cut is to run. A little dilute sulphuric acid is to be allowed to fill the groove thus made, and then a little mercury is so placed as to amalgamate the exposed metal at the scratch. If the plate is then left, with sufficient mercury in the groove, for a few hours, it penetrates the part where the grease has been removed, and renders it so brittle that a smart tap will enable the plate to be divided in the required direction.

663. The platinized silver for the inactive element may be prepared in two ways. By the first method, the silver in sheets a little thicker than writing-paper is to be roughened by a momentary immersion in ordinary nitric acid. It is then attached to a wire connected with a plate of zinc in a porous cell. In an outer cell is contained dilute sulphuric acid, mixed with a weak solution of chloride of platinum. The action is set up by weak sulphuric acid, in the same manner as in the electrotype process. The object of obtaining the platinized silver with a rough surface, is because the hydrogen gas is by this means liberated more readily. But this object is much more conveniently attained by

the following method of platinizing the silver:—A sheet of the silver is to be placed on a table between two coarse pieces of sand-paper, and the latter being strongly rubbed with a glass stopper, the silver is made to assume a very peculiar appearance, being covered with minute elevations and depressions. The silver plate thus roughened is laid upon a plate of metal as hot as may be, so that it does not scorch paper, and a dilute solution of chloride of platinum is to be applied by means of a rag: the silver will thus become thoroughly platinized and of a deep black. It is necessary that the solution of platinum should not be too strong, as it creates a tendency in the platinum to peel off from the surface of the silver.

The process of roughening by means of sand-paper, as above described, by giving an irregularly corrugated surface to the silver, greatly increases its stiffness, and thereby renders it easier to insert it in the frame than when the nitric acid method is adopted. Moreover, the surface is much better adapted to enable the bubbles of hydrogen to escape.

A strip of copper about $\frac{3}{8}$ths of an inch broad is now to be soldered to the top of the silver plate, as at *a b*, fig. 349. A frame is then constructed just large enough to allow the plate to slip between two grooves, and into another at the bottom; these serve to keep it stiff, and lessen the chances of injury. Another piece of wood, grooved in its under side to receive the copper band, *a b*, is then made; it has a hole in it to allow the binding-screw, fig. 350, to pass through so as to enable it to be soldered to the piece of copper.

Fig. 349.

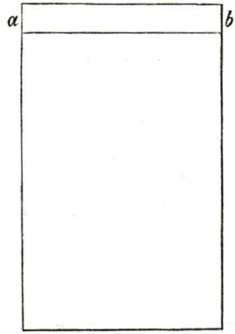


Fig. 350.

The whole arrangement of the negative element is seen in fig. 351. The binding-screw, *h*, is soldered at *c* to the copper strip *a b*. The dotted lines at *i* show the manner in which it passes through the wooden top of the frame, and those all round indicate the depth to which the platinized silver extends.

Fig. 352 gives a sectional view of the wooden frame in a plane at right angles to *d e*, fig. 351, showing the manner in which the zinc plates are attached by means of the second and large binding-screw, *a b*. The screw, *c*, is intended to enable the strips of copper, by which the connexions are made, to be tightly held. At *a* is the screw which clamps the zinc plates to the frame. The screw, *h i c*, fig. 351, is not seen in this view. The dotted lines give the direction of the plate of platinized silver. The zinc plates are seen at *d* and *e*, fig. 352.

Fig. 351.

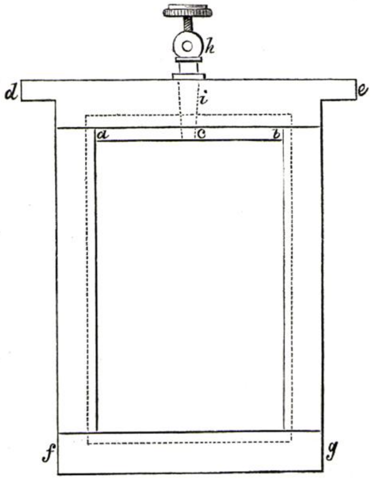
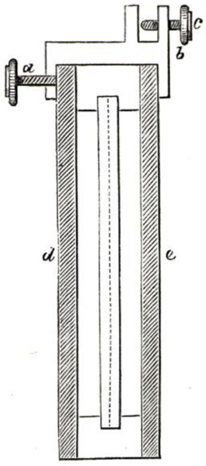


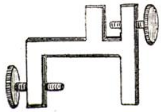
Fig. 352.



It is proper to varnish the wood-work of the battery and the copper slip, *a b*, figs. 349 and 351, with a solution of sealing-wax in methylated spirit, as it greatly preserves them from destruction by the acid. But it is essential that none of the cement finds its way on to the acting surfaces of the zinc or platinized plates, as any portion so covered is rendered useless, and no longer contributes its share to the working of the battery.

The large binding-screw, *a b*, fig. 352, is shown separately in fig. 353.

Fig. 353.



664. To excite the battery, one part by measure of sulphuric acid is cautiously added to twelve parts, likewise by measure, of water, and the mixture allowed to cool and deposit the sulphate of lead which generally precipitates from the acid on dilution.

665. The presence of nitric acid in the sulphuric acid used is to be carefully avoided, and also metallic salts must be prevented from finding their way into the exciting liquid.

Dilute sulphuric acid is without action upon amalgamated zinc, but a very small quantity of nitric acid, if present, will cause it to dissolve.

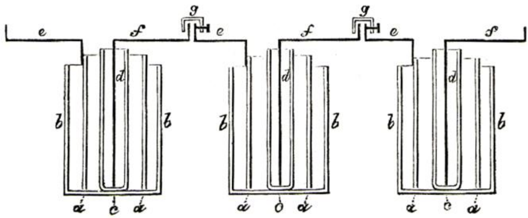
666. When it is desired to form a series of cells, it must be considered whether the experiment to be performed requires a quantity or intensity arrangement; if the former, all the zinc plates are connected together, and also all the platinized silver; this is the case where thermal effects are desired, as, for example, the ignition of a platinum wire; but if a decomposing action is required, each zinc plate is to be connected with the platinized silver of the next cell, and so on to the end of the series.

667. It is extremely convenient, where a series of plates are used, to connect them with a frame, which is slung by means of a string to a small windlass having a ratchet-wheel attached, to enable the plates to be removed out of their cells when no longer required in use.

668. *Grove's battery.*—This, the most powerful of all voltaic arrangements, may be constructed in several ways. The object being as much as possible to avoid screw-connexions in a battery evolving corrosive vapours, the method shown in fig. 354 may be adopted with advantage. Each of the stoneware cells, *b b*, *b b*, *b b*, has within it an amalgamated zinc cylinder, *a a*, to which is soldered a copper slip, *e*, bent twice at right angles. Within the zinc cylinder is placed a porous cell, *c*, containing a slip of platinum foil, *d*, to the upper end of which is soldered a ribbon of copper,

f, bent twice at right angles, like those attached to the zincs. The flat surfaces of the turned-up copper ribbons are to be opposed to each other and clamped by the binding-screws, as at *g* and *g*.

Fig. 354.



These binding-screws are of the simplest kind, and can, if required, be easily made by the operator himself. As purchased, they are made from castings, but if constructed in the laboratory, it will be more convenient to make them by turning up a piece of stout sheet-copper, as in fig. 355, and after drilling a hole in one side, making a hollow screw in it and fitting a male screw with a milled head. As it would be difficult to make a male screw with a milled head without castings for the purpose, a straight piece of metal may be soldered at right angles to the screw to serve as a purchase in turning. The apparatus for making screws will be described in the section on Miscellaneous Manipulation.

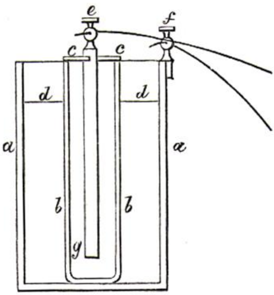
Fig. 355.

669. To excite this battery, strong nitric acid is placed in contact with the platinum inside the porous cell, and sulphuric acid diluted with seven or eight times its bulk of water is placed in the outer vessel. Some recommend only four times its bulk of water, but I prefer the strength given above for ordinary experiments. Where the platinum strips are only $2\frac{1}{2}$ inches long by 1 inch broad, and the zincs 4 inches high by $2\frac{1}{4}$ in diameter, four cells decompose water rapidly, and show the phenomena of ignition of platinum wire, &c. One such cell is strong enough for most electro-magnetic experiments, and I found six sufficient for the electrolysis of butyric acid.

670. The constant battery of Daniell, seen in fig. 356, is valuable

where it is desired to keep up a current of uniform strength for a considerable time. The outer cell, *a a*, consists of a cylinder of sheet-copper, containing within it a porous cell, *b b*, serving to hold the zinc rod, *g*, which is supported within it by means of a wooden bar, *c c*, passing through a hole pierced in the zinc. The copper cylinder and the zinc rod are provided with binding-screws, *e f*. The inner cell containing the rod of zinc is charged with dilute sulphuric acid of the same strength as that used for Smee's battery. The outer cell has a saturated solution of sulphate of copper poured into it, the strength being maintained by keeping crystals of the latter salt upon the perforated shelf, *d d*. The zinc rod must be amalgamated. When this battery is first charged, it is more powerful than it is afterwards, but after the first quarter of an hour it will remain quite uniform for several hours.

Fig. 356.



671. *Bunsen's battery.*—This form of battery, which is very much used on the Continent, has its inactive elements of carbon. They are made by heating, in properly-shaped vessels, a strongly compressed mass formed from a mixture of powdered coke and caking coal, made somewhat plastic by strong syrup. They are much improved, if immersed, after the first heating, in syrup and placed under the air-pump; on working the latter, myriads of air-bubbles rise through the syrup, and on readmitting air, the fluid enters the pores formerly filled with air. On again heating the mass to redness in a vessel well protected from the atmosphere, a fresh quantity of carbon is deposited in its pores, and the carbon becomes denser and better adapted for the required purpose. The mass wet with syrup is to be slowly but thoroughly dried before being reheated.

As it is not so easy to attach binding-screws to the carbon cylinders thus made as it is to the metal ones, a mercury-cup may be used, as will presently be described.

In the section, fig. 357, *a a* represents a porcelain pot, containing a cylinder of zinc, *b b*, to which is attached a binding-screw, *c*. Inside the zinc cylinder is placed the carbon-cell, *e*, which has the mercury-cup formed in it at *d*. The carbon-cell is filled with strong nitric acid, and the zinc is excited either with the dilute sulphuric acid previously mentioned, or a solution of common salt. The binding-screw, *c*, is represented on a larger scale in fig. 358. It is specially adapted for connexions where a ribbon of copper is used instead of a wire.

Fig. 357.

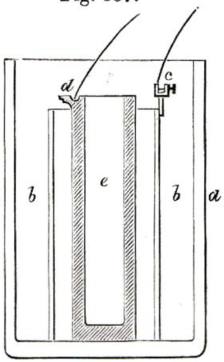
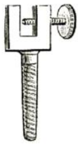


Fig. 358.



672. The above form of carbon-battery is not the only one in common use, or perhaps the most convenient. They are often constructed with the carbon element in the form of a solid cylinder intended to be placed in a porous cell of nitric acid, while the zinc element is in the form of a hollow cylinder surrounding the porous cell. Sometimes the carbon is in the form of a hollow cylinder, open at the top and bottom, and enclosing a porous cell to contain the zinc and dilute sulphuric acid; this is, perhaps, the commonest form of the Bunsen's battery imported from the Continent. In batteries of this kind the carbon has a belt of copper round its upper part fastened by a clamp. This belt enables the connexions to be made with far greater convenience than the mercury-cup.

673. The above batteries are those in common use, and are by far the most convenient: for a description of the arrangements of Faraday, Callan, Sturgeon, Wollaston, Hare, Young, Children, &c., the student is referred to works expressly treating on electricity.

Miscellaneous Galvanic Manipulation.

674. *Binding-screws and connexions.*—Several forms of binding-screws have already been described and illustrated by figures 350, 353, 355 and 358; there are, however, two others which demand attention. If, as constantly happens in experiments with batteries, two wires have to be connected, the simple binding-screw, fig. 359, may be used. It merely consists of a brass ball perforated through its centre to permit the wires to pass, and having a screw with a milled head to enable them to be compressed together. If a plate of zinc, or other metal, is to be attached to a screw for the purpose of enabling it to be placed in connexion with other pieces of apparatus, as, for example, in the single-cell electrotype arrangement, the screw, fig. 360, is exceedingly well adapted for the purpose. The slit at the lower end is slipped over the plate, and the two are made fast by means of the horizontal screw, while the vertical one allows a wire to be connected with it to support the object to be copied by the electrotype process, or it serves in other experiments to permit the positive element in one cell to be connected with the negative element in another.

Fig. 359.

Fig. 360.

675. It cannot be too strongly impressed upon the student, that all the connexions used in galvanic arrangements should be kept perfectly clean and present a bright metallic surface at the places of contact. The best forms of battery, or other galvanic apparatus, even when, as regards other matters, in the most perfect state, will fail to give good results if the connexions are dirty or have become oxidized. It is generally advisable before forming unions, to amalgamate the wires by rubbing them with protonitrate of mercury, especially where they are intended to dip into mercury-cups, as is necessary in many galvanic arrangements. Faraday recommends rubbing the wire at the place to be amalgamated with tallow and mercury applied with a piece of chamois leather, as by this means there is less tendency to tarnish than when the protonitrate is employed.

676. A very important point where substances are to be exposed to galvanic action is, that the wires which carry the electrodes should be sufficiently large to enable the current to pass with as little obstruction as possible; if, therefore, the rigidity of these thick wires is a source of inconvenience, they should nevertheless be carried as far as possible, and be continued by short ones of sufficient flexibility. The absolute terminations or electrodes are, almost invariably, of platinum, and in general it may be said that the larger they are, within certain bounds, and the nearer together, so that absolute contact is avoided, the more intense will be the action.

677. The form of apparatus in which to expose electrolytes to the action of the battery, depends upon the nature of the products; if, for example, water is to be decomposed, any of the following arrangements may be made use of; and many other fluids may be submitted to the action of the current with equal facility. It must not be forgotten, that water in a pure state offers great resistance to the passage of the electricity, and consequently is decomposed with difficulty; but the addition of a little sulphuric acid, by increasing the conducting power, greatly facilitates the action.

678. In eudiometrical experiments, it sometimes occurs that the proportion of nitrogen in the mixture is so large that it becomes impossible to explode it; when this happens, it is essential to add not only the oxygen required for combustion, but also a certain amount of oxygen and hydrogen in the proportion in which they exist in water. It is perhaps more easy to do this by electrolysing water than by any other means, as then the gases are at once obtained perfectly pure and in the proper relative proportions. In operations like this, where battery-gas is used, it is obvious, that, beyond enabling the otherwise uninflammable gas to be exploded, no other result occurs, as it totally disappears by the combustion and consequent formation of water. In experiments of the latter kind, the gases not being required separate, the apparatus, fig. 361, may be employed. The bottle, *a*, is fitted with a cork, through which two wires pass, each

having a strip of platinum foil, *b b*, attached to it to form the electrodes. These wires are connected by means of binding-screws of the form of fig. 359, with the wires of the battery, which should be stout, in order to afford as little resistance as possible to the current. The cork has a bent tube passing through it, which may be made to conduct the gas to the pneumatic trough. The bottle being filled with recently boiled distilled water, and the connexions being made, the gas evolved will be delivered into the jar previously filled with water, and inverted. When the gas is

Fig. 361.

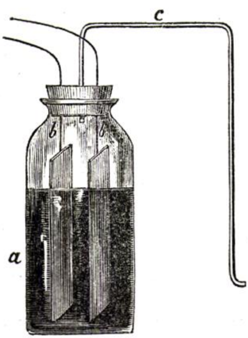
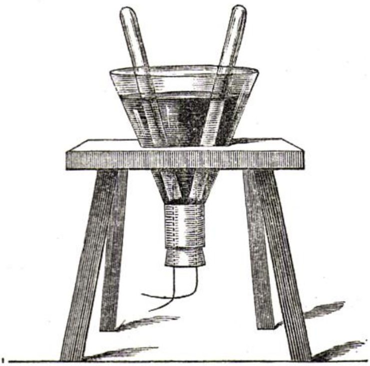


Fig. 362.



not to be used in eudiometrical researches, the boiling of the water may be omitted, and ordinary acidulated water be used instead. Of course the current must be allowed to pass until all the air is expelled, before collecting the gas.

679. If, on the other hand, it is desired to obtain the gases separate, the arrangement, fig. 362, may be adopted. A funnel is selected with a wide aperture at the lower end, and the chief portion of the tube is removed, about an inch being left. A cork made to fit accurately is cemented into its place. Two wires with platinum electrodes soldered to them are then passed through the cork. The funnel being nearly filled with acidulated water, the gas-tubes also filled with the fluid are inverted over the electrodes, and, on contact being made with the battery, the

gas ascends into them. Instead of a table with a hole in the centre, as represented in fig. 362, the apparatus may be supported on a retort-stand. This is the simplest, cheapest, and certainly one of the best modes of showing the decomposition of water.

680. If it is desired to obtain only one of the gases, as, for example, the oxygen, a tube of glass having an electrode fastened into it, fig. 363, may be filled with acidulated water, and inverted in the liquid contained in a glass or porcelain vessel, and on connecting the positive wire with *a*, and immersing the negative one

Fig. 363.

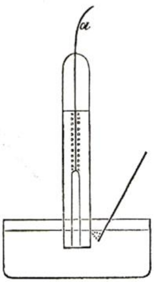


Fig. 364.

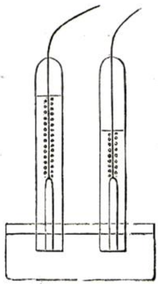
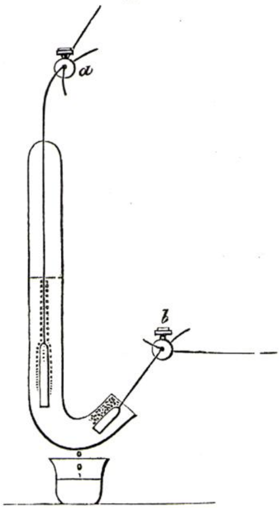


Fig. 365.



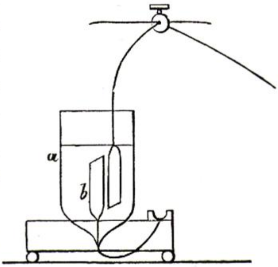
in the fluid, the oxygen will be obtained in the tube. If the hydrogen is required, it will be necessary to reverse this arrangement. Fig. 364 shows another mode of obtaining both gases in separate tubes.

681. A very neat and simple method of obtaining the gas from either electrode for examination by the use of an instrument on the principle of Cooper's mercurial receiver (§ 521), has been described by Faraday. A wire flattened out at one end is fused into a tube curved at the lower extremity, fig. 365. This is to

be filled with the fluid to be electrolysed and supported in the position represented in the figure; the other electrode is then inserted in such a manner that the gas from it does not pass into the tube, but away, as seen by the dots; that from the other electrode is thus collected in the tube, the water or other liquid falling in the tube and being collected in the vessel placed beneath. The nature of the electrodes connected at *a* and *b* of course determines the kind of gas obtained.

682. Where the object is not to collect a gas, the apparatus in the margin, which may be easily constructed, is useful. A glass tube, *a*, has a platinum wire, flattened at the extremity, *b*, fused into it; this wire may be bent up through the little stand and end in a mercury-cup. The other electrode is to be immersed in the fluid as near as may be to the other, but of course without touching.

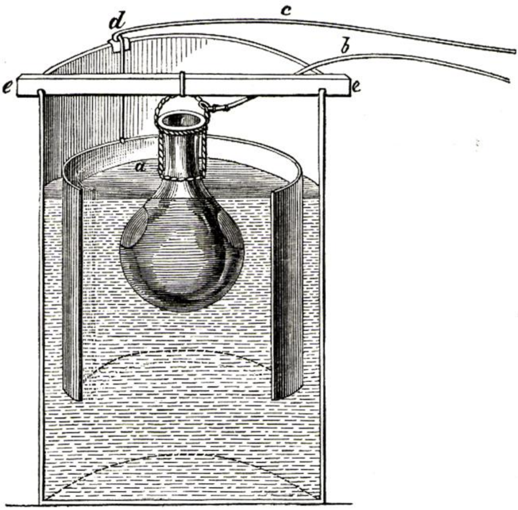
Fig. 366.



683. *Electrotype.*—There are several modes by which metals may be deposited by means of electricity on properly prepared surfaces. Almost the only application of the art which promises, at present, to facilitate chemical manipulation, is the covering of retorts and flasks with copper, by which means they are rendered less fragile, and if fracture should happen during a distillation, the contents of the retort are preserved. Moreover, the copper by its conducting property causes the flask to become hot in less time than it would without the addition; and in the distillation of fluids of rather high boiling-points, they are particularly convenient, because the coating of copper, by keeping the upper part of the distilling vessel at a high temperature, prevents condensation, and consequently accelerates the progress of the distillation.

To render the glass capable of conducting the electric current, it is first covered with a thin coating of fat varnish, except in one or two places, where spaces are to be left uncovered, in order to facilitate inspection of the contents when being used. Before the varnish has quite lost its adhesiveness, bronze powder is applied by means of a very soft brush. This covering must be quite

Fig. 367.



perfect, for wherever the glass is uncovered the copper will not be deposited. Inside a large copper pan, fig. 367, a sheet of copper coiled into a cylinder is attached by twisting, or by means of the binding-screw at *d*, with a wire connected with the battery. A bar of wood, *e e*, spans the vessel, and is notched to keep it in its place. The flask, *a*, is suspended from this bar by copper wires, and its buoyancy is overcome by filling it with water and adding sufficient shot to sink it. It is essential that the wires, which serve the twofold purpose of supporting the flask and connecting it with the battery, should be in perfect contact with the bronzed surface.

684. The flask is attached to the binding-screw of the zinc rod of a quart Daniell's battery by means of a copper wire, *b*, the cylinder surrounding it being attached to the copper of the bat-

tery by the wire, *c*. The cupreous solution to be decomposed, consists of two parts of a saturated solution of sulphate of copper and one part of a saturated solution of sulphate of soda, to which as much sulphate of copper has been added as it was capable of dissolving. The coating will become of a proper thickness in about two days.—*Redwood*. For the manipulation connected with ordinary electrotype, the reader is referred to Walker's or Napier's Treatises on Electro-metallurgy.

685. I have had occasion in some experiments to use a copper flask for the purpose of cohobating certain fluids with caustic potash. As it was necessary to have no join, I made them thus. I first modelled a flask in plaster of Paris, of the size and shape required, and from it cast a hollow mould in two pieces. An aperture of about a quarter of an inch allowed melted wax to be poured into the mould; it was then shaken round to distribute the wax equally on the interior. The excess of wax was then poured out. When perfectly cold the mould was opened, and the wax flask removed and coated externally with fine plumbago; it was then suspended inside a pan of solution of sulphate of copper, and connected with the battery in exactly the same manner as the glass flask described above. Copper was deposited for about a week, at the end of which time the metal was found thick enough, and it merely became necessary to melt out the wax and boil the flask with caustic soda. To strengthen the neck, I twisted repeated folds of copper wire round it, and again deposited copper until the wire and flask became one piece. Retorts and flasks of copper are easily made in this manner. On one occasion I constructed an alembic with moveable head by electro-deposition, and I think the method might be advantageously employed in constructing the copper apparatus required in various researches.