ferric hydroxide was added, and after digesting about one hour, the mixture was cooled and transferred to a graduated flask and diluted to 500 cc. An aliquot part of the solution was filtered off and treated by Gomberg's method. The difficulty experienced was in filtering off the periodide of caffein. The percentage of caffein obtained was 2.28, as compared with 2.29 and 2.30 by the other methods. The solution of caffein obtained by this method is not sufficiently pure for the extraction of the alkaloid by chloroform for analytical purposes.

In comparative experiments with a number of methods for the determination of caffein in teas, the Gomberg method has given the most satisfactory results and admits of wider application than the gravimetric methods.

SOME APPARATUS FOR THE TECHNICAL ANALYTICAL LABORATORY.

By Edward S. Johnson.

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THOSE branches of chemical industry which are dependent in part for guidance upon an analytical laboratory are each year becoming more exacting in regard to their demands upon the laboratory for accurate, yet rapid, work in large quantity.

Besides stimulating research with a view to shortening established methods of analysis, or the invention of new ones requiring less time for their execution, this exaction has resulted further in the introduction of much special apparatus for expediting the routine operations of the laboratory. A visit to any of the well-conducted technical laboratories of the day will show that much work of this sort has been done, and the frequent contributions on the subject in the contemporaneous journals of chemistry give evidence that activity in this direction is not abating.

It is the purpose of the present communication to present a few forms of apparatus designed in the first instance to facilitate and hasten the work of the technical laboratory with which the writer is connected. The efficient service which these devices have rendered induces him to venture upon their description with the thought that it may prove of interest to others engaged in the same field of work—the analysis of iron and steel. Although more particularly adapted to the special work mentioned, the ap-

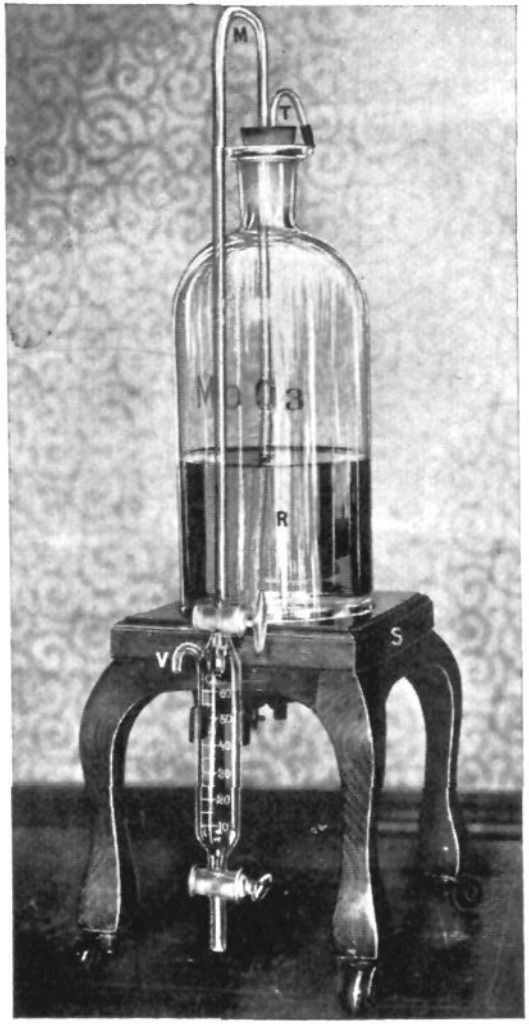

Fig. 1.

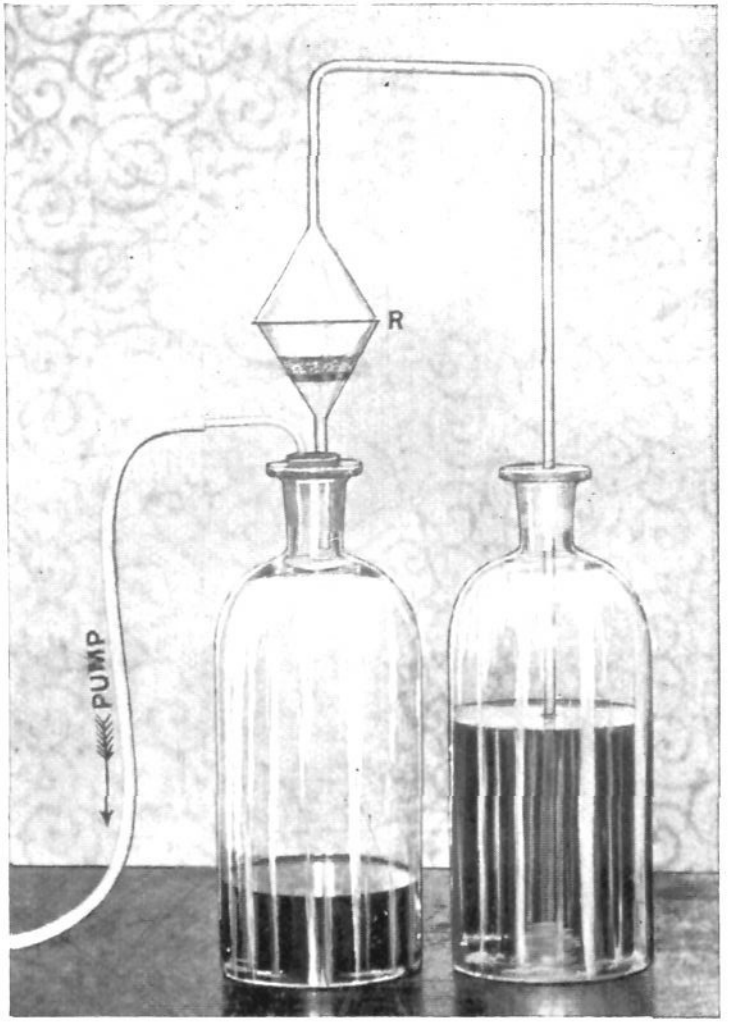

Fig. 2.

Fig. 3.

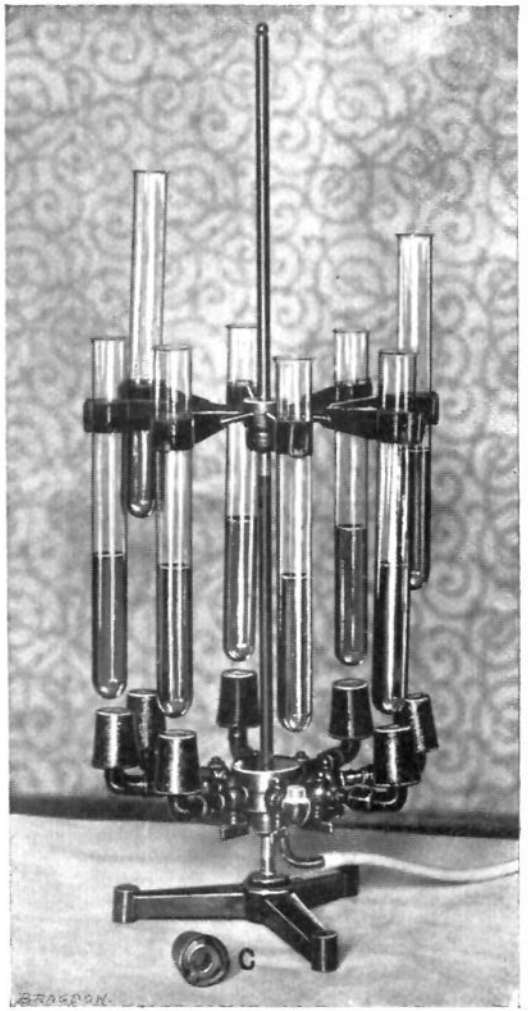

Fig. 4.

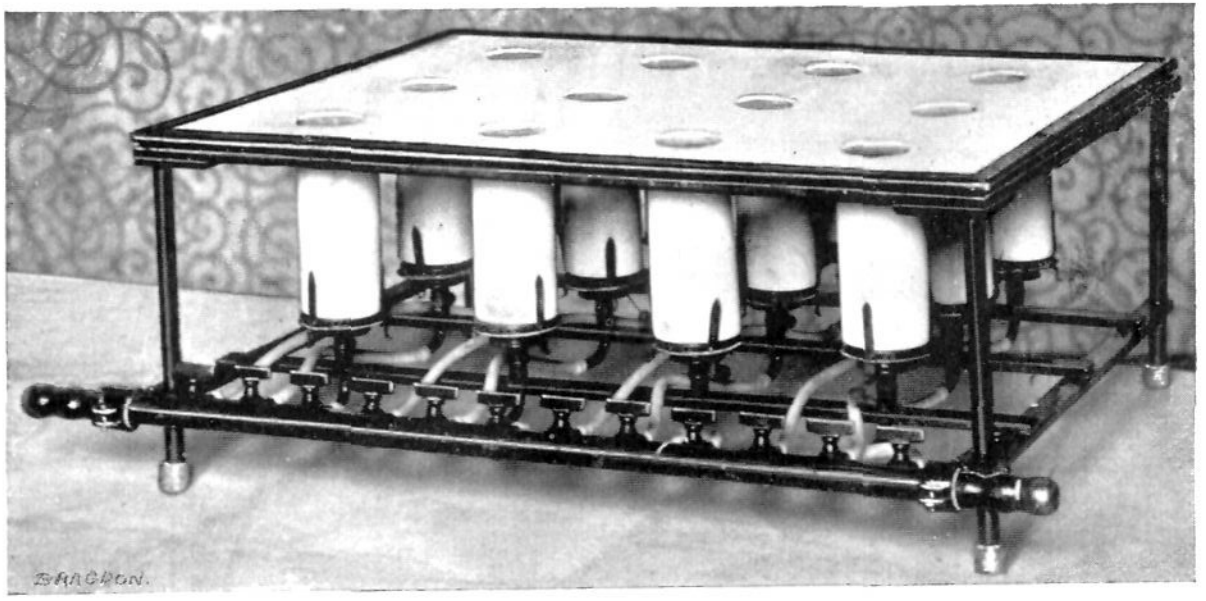

Fig. 5.

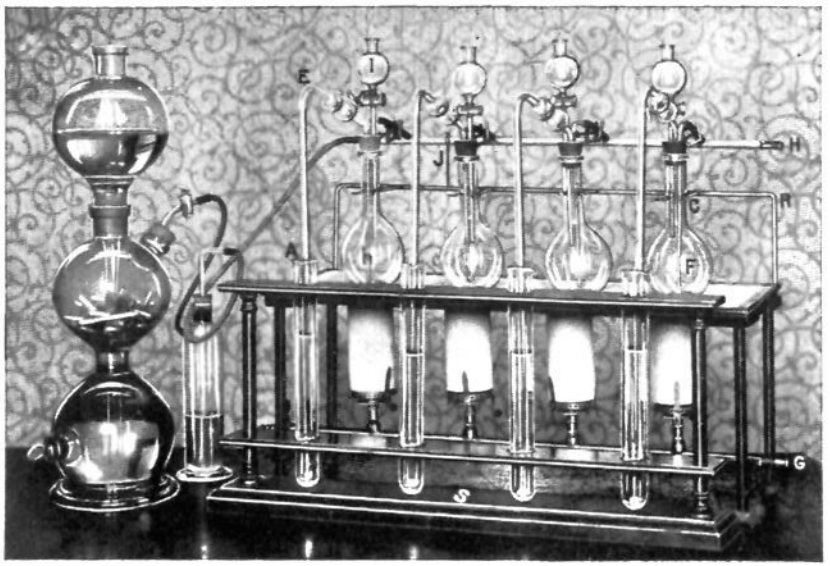

Fig. 6.

paratus, in some cases, may perhaps be found capable of advantageous application in connection with other branches of technical analysis, and possibly, in one or two instances, in the general analytical laboratory. In its application it concerns the operations of: I. Measuring Reagents; II. Filtration; III. Solution with the Aid of Heat.

I. MEASURING REAGENTS.

As is well known, the technical analyst is constantly required to treat a large number of samples or solutions successively with a given quantity of some liquid reagent in order to obtain a solution, effect a precipitation, etc., *where great accuracy is not important.* Such manipulations may be speedily executed by the simple arrangement shown in Fig. 1.

A reservoir of several liters capacity, containing the reagent, is connected by a siphon with a measure, and the whole mounted upon a stand with castors. The stand *S* is of finished oak, and ten to eleven inches in height. In its top there is a shallow circular space slightly larger in diameter than the reservoir *R* which, in this case, is a four-liter bottle. This sufficiently secures the reservoir in its position on the stand. The bottle is closed by a rubber stopper into which two glass tubes are inserted, *T* reaching to the lower surface of the stopper, and *M*, of one-quarter-inch tubing, which extends to the bottom of *R* and forms the siphon connecting with the graduate. The latter is provided at both ends with accurately ground stop-cocks. At *V* is a vent through which air escapes or enters as the graduate is filled or emptied. In the upper end of the measure and directly under the opening in the stop-cock, a short piece of tubing is fused. It directs the liquid as it enters the measure, preventing its running down the walls and consequent escape at *V*. A clamp attached to a stout wooden peg fixed in the under side of the top of the stand holds the graduate firmly.

Until the supply of reagent in the reservoir is exhausted, the apparatus is clearly ready for continuous service after the filling of the siphon. This, plainly, may be done either by applying pressure through *T* or suction at *V*, the lower stop-cock being first closed. When used it is drawn to the edge of the work-table, where the graduate projects slightly, and the reagent may be conveniently delivered into the vessels destined to receive it.

As the level of the liquid in the reservoir falls, the rapidity of the flow from the siphon of course decreases. Should the delivery become too slow, a force-pump (rubber bulb with valve) may be used to restore the failing pressure. When this addition is made, it is convenient to attach a piece of rubber tubing with a pinch-cock at *T*. It allows of quickly relieving the pressure within the reservoir. A third tube bent like *T*, but having a small bulb in the outer shank, connects with the pump. The bulb is filled with cotton which filters the air forced through the tube. Where reagents contain solids in solution, it is usually necessary, to avoid the sticking of the stop-cocks, to clean them and the graduate directly after using, especially when some time must elapse before the apparatus is again used.

In some analytical work, it is desirable to be able to deliver larger volumes (200 to 250 cc.) than is possible with the arrangement just described. This, however, is easily adapted to the work indicated. The height of the stand remains the same. The reservoir has the same capacity, or may be larger, according to the demand made upon its contents. *M* may be of somewhat larger diameter with advantage; it no longer acts as a siphon, and merely connects reservoir and measure. The measure with its increased capacity is much longer than before, and is so placed that the outer end of *M* is above the level of the liquid in the filled reservoir. The upper stop-cock may then be dispensed with, as the graduate is filled by means of the force-pump and the flow of the reagent from *M* may be instantly stopped by opening the valve (rubber tubing with pinch-cock) which is added at *T*. This and the pump, conveniences in the form of the apparatus seen in the figure, now become necessities.

By substituting for the graduate a burette, an extremely practical combination is obtained for *measuring small volumes accurately*. *M* must be made higher and dips about one inch into the burette without being attached to it. A disk of soft rubber with a hole to allow the passage of the tube, closely covers the mouth of the burette. Further, a second clamp is needed to retain the burette in a vertical position. It grasps the neck of the bottle at one end and the burette at the other, and is a simple adaptation of a familiar form of clamp. The filling of the bu-

rette, as need hardly be remarked, is conducted exactly as described for the arrangement with the larger measure.

This modification of Fig. 1 was devised for use in connection with the colorimetric determination of carbon in steel. It has so effectually aided in the work that it now seems almost indispensable. It is necessary for obvious reasons to do the work referred to in the draft-chamber. When not in use, the appliance must be kept out of the way of other apparatus and work, usually back against the wall of the chamber out of easy reach for service. The facility with which it may be moved to a convenient position when needed becomes here an especial advantage.

II. FILTRATION.

1. *Filtering Apparatus with Automatic Feed.*—For certain phases of the routine work of the technical laboratory, the necessary reagents require preparation in large quantities as an economy of time. This involves, as a rule, the filtration of solutions several liters in volume. With the device seen in Fig. 2, readily constructed from materials always at hand in the laboratory, such filtrations are rapidly executed with extreme convenience. Of the two bottles shown in the cut, the one on the right contains the unfiltered solution, while the other receives the filtrate and carries the filter noticed in the funnel held by the rubber stopper in the neck of the bottle. The filter is of thick felt formed from paper or asbestos pulp upon a perforated porcelain filtering-plate.[1] The funnel is about four inches in diameter and has a carefully ground edge. For most purposes, a plate one and a half to two inches in diameter will answer. Resting on the funnel with the filter is a second inverted one of the same diameter, also having a ground edge, and with a long stem bent to reach to the bottom of the bottle containing the solution to be filtered. A ring of soft rubber *R*, between the funnels, and their ground edges effectually secure an air-tight joint when external pressure is applied. It was first intended to use funnels with quarter-inch flanges as a means of fitting them together. The

[1] O. N. Witt: *Ber. d. chem. Ges., 1886*, 918. This exceedingly useful form of filter in the preparative work of the laboratory, particularly the organic laboratory, whether it be desired to clear a solution or collect a precipitate, has recently been used in my laboratory with advantage in certain *quantitative work*. By means of the pulp-filter, tungstic hydroxide, for instance, a most troublesome precipitate to handle in large quantities, may be collected and washed with little difficulty.

above simpler arrangement, however, as intimated, works perfectly. The end of the stem of the upper funnel has a lateral opening,[1] obviously that the solution may have unhindered entrance.

The preparation of the filter (making the pulps and forming the felt upon the plate) needs no explanation. When asbestos is used it should first be ignited. The fibers then become somewhat elastic and produce a spongy, rapid filter. The felts are usually three-eighths to one inch thick, according to the difficulty of the filtration, the thicker felts being used for the more obstinate cases.

The filtration is begun, after placing the parts of the apparatus as seen in the figure, by starting the filter-pump. The bottle to receive the filtrate and the space enclosed by the funnels, as a result, are partially exhausted of air. The external air-pressure now being considerably greater, the funnels are pressed forcibly together. The solution to be filtered rises and flows upon the filter, the flow continuing only as fast as liquid is withdrawn; the solution is thus automatically fed to the filter. The apparatus requires no further attention until the filtration is complete.

2. *A Revolving Filter-Stand.*—This device is represented by Fig. 3. The tripod base carries a vertical steel rod about three-eighths of an inch in diameter and eight inches high. Its free end is rounded and forms the pivot for the rest of the apparatus. This consists of a central column of brass and two disks of oak, each of three crossed layers. The column fits closely over the steel rod, and is one inch in diameter and fourteen inches high. On its lower end is a broad flange to which the larger disk is attached. The disk is nineteen inches in diameter and one inch thick, including the rim which projects about one-quarter of an inch. The upper disk is ten and one-half inches in diameter and one-half inch thick. It contains fifteen tapered holes on the circumferences of an outer and inner circle, twelve in the outer and three in the inner. The holes are about one and one-quarter inch in diameter on the upper side of the disk. Screwed to its under side is a flanged brass collar with set-screw, by which its height on the column may be adjusted. Both disks are well finished and protected against moisture by outside varnish.

[1] Made by blowing a bubble in the tube at the desired point, rubbing it off, fusing the edge of the opening left, and cutting the tube through the center of the hole.

When in use, sheet rubber covers are placed on them as a further protection. At *S* is a swivel with a clamp. It serves to hold the rubber tube through which liquid is supplied from an elevated reservoir for washing the precipitates and residues collected upon the filters. Through a small hole in the swivel at the base of the holder, the steel rod bearing the column and its disks may be oiled.

The stand, as described, was designed especially for handling the large number of ammonium phosphomolybdate precipitates obtained from the phosphorus determinations of the steel works' laboratory. The beakers are No. 1 Griffin's and the funnels two inches in diameter. Three of the fifteen beakers with which the stand may be used are placed upon the upper disk during the filtration.

III. SOLUTION WITH THE AID OF HEAT.

1. *Solution in Large Test-Tubes.*—In Fig. 4 is shown an apparatus[1] devised for use in the colorimetric determination of manganese in steel. The part of the process here concerned involves the solution of the sample in nitric acid, heating the solution to boiling, and the oxidation of the manganese it may contain to permanganic acid by continuing the boiling after the addition of lead peroxide. The samples are dissolved in ten-inch by one-inch test-tubes, eight solutions being made at once. The tubes are supported in a vertical position by a set of equi-distant clamps, modifications of a well-known form of spring-clamp. They are of stiff sheet brass, as regards the jaws, and their shafts screw into a central cylindrical block of the same material. The whole revolves about the three-eighth-inch vertical brass rod of the tripod-stand, and is supported by a similar block which may be fixed at any point on the rod by the usual set-screw. Below the clamps are eight Bunsen burners, radiating at angles of 45° from a short hollow cylinder which distributes gas to the burners. Their position on the axis is also adjustable. The burners are provided each with a stop-cock and chimney. The manner of attaching the chimney is shown at *C*. To protect the apparatus from the corrosive vapors to which it is constantly

[1] Made by Messrs. Bullock & Crenshaw, of Philadelphia, to whom credit is due for the careful execution of the idea of the apparatus as conveyed in a somewhat crude sketch prepared during the hurry of remodeling and re-equipping a laboratory. The heating apparatus shown in Fig. 5 was also constructed by the same firm.

exposed, it is varnished with special care: the polished brass parts with coach-varnish, the iron base and copper chimneys with asphaltum varnish.

By the above construction and adjustments, the solutions in the test-tubes may be brought almost simultaneously to boiling, and further treated as already pointed out. To arrest the boiling of the solutions, a slight movement of the set of clamps to the right or left suffices; if it be desired to remove only a part from the action of the flames, the tubes are drawn up in the clamps as shown in two instances in the cut.

2. *Solution in Dishes, Beakers, and Flasks.*—A few remarks of explanation in connection with Fig. 5 will make plain the construction of the apparatus here referred to. A rectangular iron stand nine inches high, twenty-five inches long, and eighteen inches wide forms its framework. This is built of an upper and lower frame, joined by legs of half-inch round bars. Low down on the legs is the lower frame, consisting of two end-pieces through which the legs pass, and three similar bars at right angles to the end-pieces. The bars of the frame are about one inch by three-sixteenths in size. Its parts are substantially riveted together. Within slight limits it may be given a higher or lower position. Screwed into each of the longer bars are four Argand burners with four-inch clay chimneys. The burners are connected by rubber tubing with the stop-cock of the one-inch brass pipe attached in front to the legs of the stand. The manner of connecting the tubing with the burners is best seen in the case of the middle row. Through the pipe in front, from either end, and its stop-cocks, gas may be supplied to any one, part, or all of the burners. The first, fourth, seventh, and tenth stop-cock, counting from the left, connect with the rear row of burners; the second, fifth, eighth, and eleventh with the middle row, etc.

The upper frame, above the burners, bears a loose asbestos pad, which forms the top of the stand. It is rimmed with sheet copper and has twelve two-inch holes, each directly over a burner. Two cross bars in the frame, parallel to those in the lower frame, prevent a sagging of the pad.

The feet of the stand are shod with rubber (stoppers bored partially through with a cork-borer) for an apparent reason.

To subserve the occasional cleaning and revarnishing to

which the apparatus, used as it is in the corroding atmosphere of the draft-chamber, must be subjected, the burners, as implied above, can be easily removed, and the gas-pipe in front detached from the clamps which support it. Besides hastening solution, this device may also be used with advantage for certain evaporations and other operations which at once suggest themselves.

The compactness of the apparatus, the ease with which the flames are perfectly controlled, due to the position of the stop-cocks, and a definite plan in their connection with the burners, the varied application of which it is capable, and its convenient portability combine to make it a most useful appliance in the laboratory.

3. An Apparatus for Use in the Determination of Sulphur in Steel by the Evolution Method.—Although excellent forms of apparatus have been proposed for the purpose indicated, a description of the combination shown in Fig. 6 is nevertheless undertaken, it seeming to the writer to have advantages which apologize for its addition to the already numerous list which might be compiled from the literature of the subject.

That part of the familiar method for the determination, which the present apparatus was designed to execute, consists in dissolving the sample of metal in hydrochloric acid, and the passage of the gases formed through an ammoniacal solution of cadmium chloride. The contact of acid and metal results at first usually in a brisk evolution of gas. This, however, soon slackens, and is then restored to the desired moderately rapid rate by the application of heat. As the metal dissolves, heating fails to maintain a steady flow of gas. At this stage of the process, hydrogen is forced into the flask in which the solution is now nearly completed, and next heated to boiling. The hydrogen serves (as need scarcely be remarked) a double purpose: the residual gases in the flasks are driven rapidly into the cadmium solution, and the liability (especially when the attention of the analyst must be divided among a number of simultaneous determinations) of the latter's "striking back" into the flask, during the boiling, is entirely obviated.

The following references to the figure will make plain the relation of the parts of the arrangement shown by it to each other and the manipulations thus roughly sketched.

The 250 cc. flasks *F*, in which the metal and acid are brought in contact, stand upon a light iron table nine inches high, seven inches wide, and twenty-three inches long. The top of the table is of asbestos board as in Fig. 5; under each flask, there is a two-inch hole through which, obviously, heat is applied to the flasks from the burners below. A stop-cock at one end of *G* enables the regulation of the supply of gas to all the burners. It is, however, essential for apparent reasons, to be able to perfectly control the supply to each burner separately. This has been effected, not by the use of the customary stop-cock, but by a slight modification of the interior mechanism of the burner. This may be seen by unscrewing the stem at the milled ring. In the center of the circle which forms the inner end of the stem, is an eighth-inch opening, and at the circumference is a pin-hole, both for admitting gas to the crown of the burner. When the parts (stem and crown) are in place, a quarter-inch disk faces the end of the stem, and is adjustable, in regard to its distance from the end, by means of the threaded shaft to which it is attached or, more accurately, of which it is a part. The shaft itself is shifted toward or from the end by the key underneath the crown. The motion toward the end at its limit brings the disk in contact with it, and more or less perfectly closes the larger opening; the pin-hole being beyond the circumference of the disk remains open.

This construction does not admit of the required perfect control of the gas-supply to the individual burner. The smallest quantity of gas that may enter produces generally too large a flame when little heat is needed, and the pin-hole prevents entirely cutting off the supply. This difficulty is resolved and the burner completely adapted to the present purpose by merely closing the pin-hole and placing a circular washer of soft leather, with a small segment removed, on the disk. If the crown and stem be now screwed together, the washer is compressed between the disk and the end of the stem, tightly closing the remaining opening in the latter. Unscrewing the parts but slightly, relieves the pressure and renews communication between the stem and the crown. The manner of manipulation with the burner thus modified is plain. Additional comment is unnecessary.

The pipe *G* is strapped to the flat bar supporting it, by strips of brass fastened with screws, and is therefore readily detached. The bar is attached to the legs of the stand in the manner described for those carrying the burners in Fig. 5, and is also adjustable as regards its level. Four spring-clamps projecting horizontally from the brass rail *R* hold the flasks securely in their places. The rail is screwed to the stand underneath the top frame. *R* further has two vertical clamps *J* similar to those just mentioned. They grasp the glass tube *H*, which is closed at one end by a rubber cap and connected at the other with a hydrogen-generator. Through *H* hydrogen is distributed to the flasks. For this purpose there is a short branch opposite each flask. The neck of the flask is closed by a rubber stopper, through which pass three glass tubes. One of these, *h*, is connected by rubber tubing with the branch to which attention was called, and reaches to the bottom of the flask. The lower end is drawn out and bent upwards slightly. By means of a compressor, the rubber tubing making the connection may be opened or closed as desired. The dropping-funnel *I*, for introducing the acid, has a wide-mouthed bulb large enough to contain the whole of the acid required for the solution; the stem holds a column of acid capable of more than overcoming the resistance offered by the solution in the cadmium chloride tubes, the ten by one-inch lipped test-tubes, *A*. The tube *E* with two bulbs conducts the gases generated in the flask into the cadmium solution in *A*. The test-tubes stand, in the rack *S*, upon a pad of sheet rubber.

Economy of space, the ease, nicety, and rapidity of manipulation possible with it, and the ability to use the device wherever the little space occupied by it may be found in the draft-chamber, are its practical features. Where a large number of determinations must be made at one time the number of stands may be correspondingly increased and controlled by one operator.

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