the ionization of the mercuric chloride until it becomes lower than that of the mercurous chloride (formed by the reducing action of stannous chloride), and then the latter will dissolve, forming mercuric chloride and mercurous sulphate. Since mercurous sulphate exerts a reducing action on permanganate, this will introduce an error into the determination. Our experiments have shown that anywhere from twenty-five to fifty cc. of mercuric sulphate solution to ten cc. of hydrochloric acid, will give accurate results in the presence of small quantities of mercurous chloride.

The points requiring special care in this process then are :

1. Use stannous chloride that is in good condition.

2. Have the smallest possible excess of stannous chloride present after the reduction is completed.

3. Carefully adjust the proportions between the mercuric sulphate and the hydrochloric acid.

The speed of this process leaves nothing to be desired. Less than three minutes, in the case of soluble salts, has been found sufficient time for solution, reduction, and titration.

In conclusion we wish to acknowledge our indebtedness to Prof. E. H. S. Bailey for his advice and many valuable suggestions during the progress of the work.

A NEW APPARATUS FOR SULPHUR DETERMINATIONS IN IRON AND STEEL AND A USEFUL FORM OF WASH-BOTTLE.

BY RICHARD K. MEADE.

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THE writer has found the two forms of chemical apparatus mentioned below of much use in his work and describes them here, in the hope that they may prove of equal service to other analysts. The first description is that of an apparatus to be used in the determination of sulphur in iron and steel, by the method of conversion into hydrogen sulphide and absorption of the evolved gas, in an alkaline solution of lead nitrate.

The apparatus is shown, drawn to one-eighth scale, in Fig. 1. It consists of the following parts :

1. A half liter flask, of the "Joliet" pattern, such as is used in the laboratories of iron and steel companies. The flask is

fitted with a rubber stopper, having two holes, through one of which passes a funnel with glass stop-cock, and through the other a piece of glass tubing, bent at right angles about its middle point.

2. A one-ounce separatory funnel, cylindrical in shape, fitted with a two hole rubber stopper, provided with tubes for the admission and exit of the gases, as shown in the cut. The

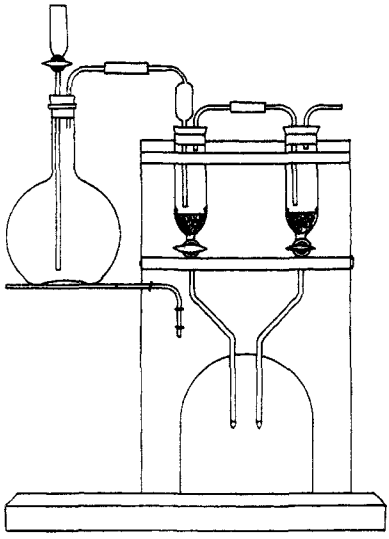

Fig. 1.

delivery tube is blown into a bulb of twenty-five to fifty cc. capacity, in order to prevent any of the solution in the funnel from being sucked into the evolution flask. The funnel itself is dented in three places, the same distance and about one-half inch from the bottom of the funnel, or just where the walls begin to narrow to meet the stop-cock. This may be easily done by cautiously heating the funnel in the flame of a blast-lamp and then pressing lightly at the desired points of indentation, with an iron wire. In the bottom of the funnel is placed a little glass wool, and upon this sufficient asbestos to reach even with the dents in the walls of the funnel. Over this a thin perforated disk of platinum, made to fit the funnel closely, is pressed down under the dents, holding the asbestos in place. The stems of the funnels are bent as shown in the sketch.

3. A separatory funnel, fitted similarly to the one described above, except that the tube leading into it need not be blown into a bulb.

4. A wooden base or stand upon which the whole rests. It is made exactly as shown in the sketch. The guide or upper cross piece is one and three-fourths inches wide and made solid. The rest, or lower one, is the same width, but is split along its middle, hinged at one end and fastened at the other by a catch, to allow the removal of the funnels when desirable. The iron ring, upon which the flask rests, is forged of one-fourth inch wrought iron and is fastened to the stand by means of staples.

The solution of the drillings, the preparation of the absorbent solution, and the precipitation of the sulphur as barium sulphate is conducted according to Blair, as described in his admirable book, "The Chemical Analysis of Iron."

The separatory funnels are half filled with an alkaline solution of lead nitrate, the sample weighed into the flask, the apparatus connected together and dilute acid poured upon the drillings. When action slackens, a lamp is placed under the flask and its contents boiled until all gas is given off. Pass a moderately rapid current of hydrogen through the apparatus for a few minutes and then disconnect the flask and funnels. Open the stop-cocks to the separatory funnels and allow the alkaline solution to run into a beaker, previously placed under them. The lead sulphide is caught by and retained upon the filters. Wash off the tubes into the funnels and pass a stream of water, from a wash-bottle, around the sides of the funnels several times, allowing the wash water to run through the filter into the beaker. Close the stop-cocks, remove the beaker under the funnels and put a clean one in its place. Drop a little powdered potassium chlorate, free from sulphur, into the funnels, and then add ten to twenty cc. of concentrated hydrochloric acid. Stand the apparatus in a hood, or where the fumes will be carried off readily, and open the stop-cocks of the funnels, allowing the acid to drop slowly into the beaker. As it passes through the filters it dissolves any sulphide which may be mixed with the asbestos. Wash the funnels and filters well with water, allowing the washings to run into the beaker. Heat the solution to boiling, nearly

neutralize with ammonia and precipitate the sulphur with barium chloride.

It very seldom happens that all the hydrogen sulphide is not absorbed by the solution in the first funnel, so that generally it is only necessary to collect and dissolve the precipitate formed in the first funnel; should any form, however, in the second funnel, this must be collected and dissolved, and the solution added to that from the first funnel. The solution in the second funnel may be used over for many analyses.

It is hardly necessary to caution chemists against allowing the solutions to stand for any great length of time, in the funnels, on account of their action upon the glass stop-cocks. The filters in the bottom of the funnels, if properly prepared, will last for many operations.

The apparatus may be used for the determination of sulphur by the method of absorption in cadmium chloride solution, with subsequent solution of the cadmium sulphide in hydrochloric acid and titration of the liberated hydrogen sulphide with iodine, or it may be used for determining sulphur by the Elliott method, or Drown's method of absorption in potassium permanganate solution. In either of the last two cases, the filters in the bottom of the funnels should be left out, since there is no need for them. The writer has found it much easier to wash out the funnels than U tubes and potash bulbs, thus saving time and trouble and lessening the chance of error.

Many other uses will suggest themselves to the reader, to which the apparatus, modified in one way or another, may be put.

Fig. 2 represents a wash-bottle which the writer has had in constant use for some time and found very convenient. Its construction is so simple and evident from the drawing, that a description seems almost unnecessary. It consists of an ordinary wash-bottle, fitted up as usual, except that the shorter tube is made of heavy-walled glass tubing. To this is fastened a piece of hard wood, three-eighths by one-quarter by one and three-quarter inches, notched with a V-shaped notch across its face and, with a groove cut down its either side, parallel to the neck of the flask. A sketch showing the piece of wood, in detail, is appended to the drawing. A piece of stout wire is bent

with a loop at either end. The larger of these loops is made of sufficient size to admit the thumb comfortably and is covered

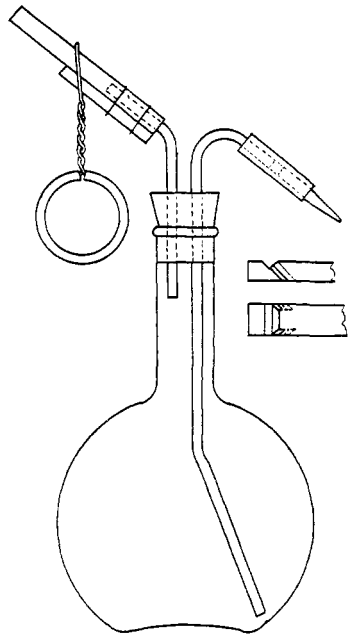

Fig. 2.

with a piece of small rubber tubing to prevent chafing that member. The smaller is bent over the rubber tubing and wood and is large enough to slide up and down freely in the vertical grooves, with about half an inch play. The bottle is grasped by the last three fingers of the hand and the thumb is passed through the loop in the wire, leaving the first finger free to guide the jet. A slight pull from the thumb is sufficient to bring the wire down upon the rubber, pressing the latter into the notch and effectually closing the opening. By doing this, before ceasing to blow into the bottle, the fumes of the acid, or steam, or whatever volatile substance the bottle may contain, are prevented from passing back into the mouth or lungs and a steady stream is kept going from the jet for some moments, or until the pressure of the thumb is relaxed, without further effort on the part of the operator.

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