3. The ether separation removes practically all the iron from the titanium very quickly.

4. Refusion of the ignited product with sodium carbonate for purification, which requires considerable time, is never necessary by use of this method. The final product is pure, unless zirconium is present, in which case this element is removed by the usual phosphate precipitation.

5. This method combines the colorimetric and gravimetric determinations and the color comparison is made in solutions always totally free from iron. For convenience hydrochloric rather than sulphuric acid solutions of titanic acid are used.

6. The method is accurate and not long.

7. Rosenheim and Schütte's yellow compound probably owes its color to the presence of peroxides in the ether used.

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APPARATUS FOR THE DETERMINATION OF ARSENIC.

BY OTIS D. SWETT.

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The many existing forms of apparatus for the determination of arsenic which give trustworthy results appear to have left little to be sought for with regard to this most essential feature. The more or less complex construction of these forms, however, together with the sources of error which usually accompany such construction, seem to justify an attempt to devise an apparatus for arsenic determination which, besides yielding accurate results, shall comprise fewer parts than are found in the other forms, and shall be compact and portable.

With these points in mind, an apparatus has been devised which is illustrated in the accompanying drawing.[1]

A is a round wooden base with a central depression and three vertical lugs which form an extension of the sides of the depression.

B is a glass vessel, 4 cm. outside diameter and 14 cm. long, with flat closed bottom, supported by the base *A* in the depression between the vertical lugs. The vessel *B* is provided with a lower side tube *b*, projecting beyond the base *A* between the vertical lugs, and with the upper side tube *b'*.

C is an inner tube, 18 mm. outside diameter, closed at its lower end, enlarged conically at its upper open end, rolled out and sealed to the top of the vessel *B*, which is closed in to meet it, and then ground on its inner surface to receive a stopper.

D is a hollow stopper with dome-shaped closed top, tapering to the other end, which is open, and ground to fit the neck of the inner tube *C*.

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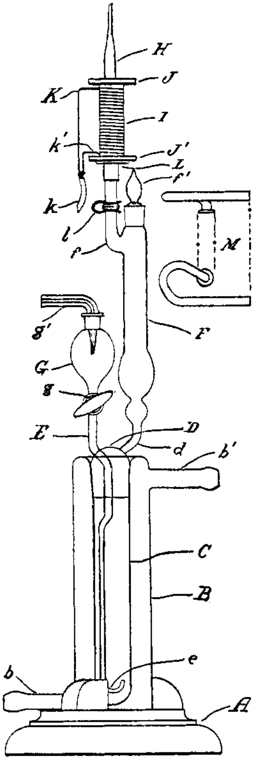
[1] Glass work by Mr. Edw. O. Sperling, Bureau of Standards.

F is a vertical drying tube, with the usual enlargement and moisture bulb, communicating with the interior of the stopper *D* through the offset tube *d*.



f is a side tube, extending laterally from near the top of the drying tube to the vertical axis of the apparatus, then vertically to the top level of the drying tube, flattened and ground to fit the correspondingly flattened and ground ignition tube *H*.

f' is a light ground glass stopper fitting the ground open end of the drying tube.

E is a tube passing through, and sealed to, the top of the hollow stopper *D*, and extending within the inner tube *C* to near the bottom, where it terminates in a gas trap, *e*, and extending upwards from the hollow stopper *C*, with an offset bend, to the stopcock *g* and communicating through this with the 5 cc. funnel *G*, which is closed in at the top to a small neck ground to take the enlarged and suitably ground capillary tube *g'*, this latter being drawn out somewhat smaller at the lower end, bent at right angles above the funnel neck, and so shaped at the outer end as to accommodate a rubber tubing.

H is a hard glass tube 6–4 mm., drawn out at one end in a suitable manner for the production of the mirror, and flattened and ground at the other end to fit the similarly finished end of the side tube *f* as described.

I is a heating coil, consisting of a threaded spool of "lavite"[1] 12 mm. outside diameter and 8 mm. inside diameter, wound with number 26 "Advance" resistance wire,[2] and mounted with a shield disc *J*, of the same material which prevents the tube above from becoming overheated.

J' is a "lavite" disc similar to *J*, upon which the heating tube *I* rests.

K, *K'* are the conducting wires and *k* is the two-wire insulated conductor from which they project.

L is a metallic sleeve, fitting snugly over the ignition tube *H*, cut to form spring fingers bearing firmly upon the ignition tube, and carrying, as an integral part, a disc which forms a flange around the upper end of the sleeve, thereby providing a means for supporting and vertically adjusting the heating coil *I*.

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[1] Manufactured by the D. M. Steward Mfg. Co., Chattanooga, Tenn.

[2] Manufactured by the Driver-Harris Wire Co., Harrison, Newark, New Jersey.

l is a clip, adapted to hold firmly together the flattened and ground ends of the side tube *f* and ignition tube *H*.

M is a glass tube 5 to 7 mm. inside diameter made to fit the flattened and ground end of the tube and bent horizontally to keep within the radius of the apparatus.

The ignition tube first employed was of transparent silica, mounted by a short mirror tip. The metal clip being above the heating tube, and not thoroughly shielded from the heating coil, gave rise to irregular mirrors. When a tube, however, is used for both ignition and deposition, a number of such tubes must be on hand when making up a set of standards and for consecutive analyses, so that silica, on account of its cost, is barred. Instead, Jena boro-silicate glass, or indeed a high quality combustion glass, may be used.

A modified form of the vessel *B* has been designed, which is essentially an Erlenmeyer flask, provided with side tubes and carrying the sealed-in tube *C*, thereby eliminating the wooden base, and providing space for a larger reaction vessel.

With slight modifications this apparatus is adapted for use with many methods heretofore proposed.

For the Marsh,[1] Berzelius-Marsh,[2] and Sanger-Berzelius-Marsh[3] methods, the drying tube is charged with calcium chloride retained in place by means of absorbent cotton plugs. Lump or granulated arsenic-free zinc is introduced into the tube *C*, sulphuric or hydrochloric acid is admitted from the funnel *C* through the cock *g* and tube *E*, followed by the solution to be tested for arsenic after all air has been expelled from the apparatus.

In the application of the Gutzeit, Ritsert, Flückiger, Lohmann[4] methods, applied quantitatively by means of a set of standards for comparison, the drying tube is charged with absorbent cotton and a few strips of lead acetate paper or its equivalent, and the ignition tube is replaced with the curved tube *N*, into which the impregnated test paper is introduced.

The stopper tube *g′* is provided for connection with a supply of arsenic-free hydrogen, as suggested by Munroe and in use by his students,[5] thereby materially reducing the time required for a determination by affording a means of rapidly removing all air from the apparatus, and also effecting a saving of arsenic-free zinc and acid.

Provision is made by the outer vessel or jacket *B*, with the side tubes *b* and *b′*, for either cooling the reaction chamber *C*, as recommended by

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[1] Edin. Philos., 16, 229.

[2] *Berzelius*, *Jahresb.*, 17, 191.

[3] *Am. Chem. J.*, 13, 431.

[4] Fresenius, "Qual. Analysis," 3157, 7, e. f.

[5] THIS JOURNAL, 17, 885, 890 (1895).

the earlier authorities, or for heating it as recommended by Harkins,[1] by passing through it either cold or hot water.

In the preparation of standard mirrors for comparison, as recommended by Sanger,[2] as small a quantity as 0.005 mg. As_2O_3 may be standardized by means of this apparatus. The mirrors prepared vary distinctly with a variation of 0.005 mg. of As_2O_3 in the known solutions used and duplicate mirrors from equal quantities of As_2O_3 agree quite as well as those illustrated in Sanger's paper.

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A COLORIMETRIC DETERMINATION OF MANGANESE IN THE PRESENCE OF IRON.

BY M. R. SCHMIDT.

Received June 17, 1910.

The following method has been used by the writer for some time, and has been found to give uniform satisfaction in the determination of small quantities of manganese in the presence of iron, in certain pharmaceutical preparations, such as peptonates, sirup of hypophosphites, etc. No originality can be claimed for the method, as it was proposed several years ago by Walter,[3] but it is believed that the details of manipulation here given will lead to the greatest rapidity in attaining accurate results The usual basic acetate method of separating manganese from iron requires, in order to effect complete separation, at least two precipitations of the iron, and subsequent evaporation of the filtrates in order to insure complete precipitation of the manganese by bromine or other reagents. This involves considerable loss of time, and when dealing with small quantities of manganese, the total experimental errors often amount to a considerable percentage of the weight of the manganese involved. The present method requires no previous removal of iron, and the results are accurate when the total manganese amounts only to a few milligrams Moreover, duplicate determinations can be made in one-half hour. The determination depends on the power of the persulphates to convert bivalent manganese into permanganic acid in the presence of silver nitrate.

A standard solution of manganese containing 2 mg. of manganese in each 10 cc. is found to be most convenient, and is prepared as follows: Run 182.1 cc. of exactly tenth-normal potassium permanganate solution into a liter flask, add about 100 cc. of 10 per cent. sulphuric acid and 12 cc. of 3 per cent. hydrogen peroxide. Warm the flask slightly until the oxygen is driven off, cool again and dilute to the mark. Prepare also a

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[1] THIS JOURNAL, 32, 520 (1910).

[2] *Loc. cit.*

[3] *Chem. News*, 84, 239 (1901).