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“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF COPPER

(***Revised.***)

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF COPPER (*Revised*)

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CONTENTS

	PAGE
0. FOREWORD	4
1. SCOPE	5
2. SAMPLING	5
3. QUALITY OF REAGENTS	5
4. DETERMINATION OF COPPER BY THE ELECTROLYTIC METHOD	6
5. DETERMINATION OF ARSENIC AND ANTIMONY BY THE CO- PRECIPITATION (IODIMETRIC) METHOD	9
6. DETERMINATION OF ARSENIC BY THE HYPHOSPHITE IODI- METRIC (ALTERNATE) METHOD (APPLICABLE IN ABSENCE OF Se and Te)	12
7. DETERMINATION OF BISMUTH BY THE IODIDE (PHOTOMETRIC) METHOD	14
8. DETERMINATION OF IRON	16
By the Dichromate (Volumetric) Method	17
By the Ammonium Thiocyanate (Photometric) Method	18
9. DETERMINATION OF LEAD BY THE COLORIMETRIC METHOD	19
10. DETERMINATION OF NICKEL BY THE DIMETHYLGLYOXIME (GRAVIMETRIC) METHOD	21
11. DETERMINATION OF SELENIUM BY THE GRAVIMETRIC METHOD	21
12. DETERMINATION OF TELLURIUM BY THE GRAVIMETRIC METHOD	23
13. DETERMINATION OF OXYGEN BY THE COMBUSTION METHOD	23
14. DETERMINATION OF TIN BY THE IODIMETRIC METHOD . . .	26
15. DETERMINATION OF PHOSPHORUS BY ALKALIMETRIC METHOD	28

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF COPPER *(Revised)*

0. F O R E W O R D

0.1 This revised Indian Standard was adopted by the Indian Standards Institution on 28 January 1964, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 This standard was originally published in 1955 and covered the chemical analysis of copper as specified in IS : 191-1950 Specification for Copper. In the revised version of IS : 191-1950, which was published in 1958, and its subsequent amendment published in 1961, a few more grades have been included and the chemical composition modified. Necessity was, therefore, felt to revise this standard also so that it could cover completely all the elements within the chemical composition ranges as specified in *IS : 191-1958 Specification for Copper (*Revised*).

0.3 In this revised version, changes mentioned in 0.3.1 to 0.3.3 have been introduced.

0.3.1 The following methods have been added:

- a) Determination of Arsenic by the Hypophosphite Method, and
- b) Determination of Iron by the Volumetric Method.

0.3.2 The colorimetric methods for bismuth and iron given in IS : 440-1955 have been replaced by the photometric methods.

0.3.3 The methods for determination of copper, tin, nickel, arsenic, antimony and lead given in IS : 440-1955 have been revised.

0.4 The methods of analysis prescribed in this standard have been prepared with a view that they shall be useful as referee methods. Due consideration has been given in the preparation of this standard to the facilities available in the country for such analysis.

*Second revision issued in 1967.

0.5 Taking into consideration the views of producers, consumers and testing authorities, the Sectional Committee responsible for the preparation of this standard felt that it should be related to the technological methods followed in the country in this field. Assistance has been derived from the following publications:

1962 **BOOK OF ASTM METHODS FOR CHEMICAL ANALYSIS OF METALS.**
American Society for Testing and Materials.

B.S. 1800 : 1951 **METHODS FOR THE ANALYSIS OF RAW COPPER.**
British Standards Institution.

FURMAN, N. H. (ED.) SCOTT, W. W. **Standard Methods of Chemical Analysis.** 5th ed. New York. D. Van Nostrand Company, Inc., 1962.

HILLEBRAND, W. F., LUNDELL, G. E. F. and **BRIGHT, H. A.** **Applied Inorganic Analysis,** 2nd ed. New York. John Wiley and Sons, 1955.

0.6 Wherever a reference to any Indian Standard appears in this standard, it shall be taken as a reference to the latest version of the standard.

0.7 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960 **Rules for Rounding Off Numerical Values (Revised).**

1. SCOPE

1.1 This standard prescribes the methods for determination of copper, arsenic, antimony, bismuth, iron, lead, nickel, selenium, tellurium, oxygen, tin and phosphorus in the ranges as specified in *IS: 191-1958 Specification for Copper (*Revised*).

2. SAMPLING

2.1 Samples shall be drawn in accordance with IS : 1817-1961 Methods of Sampling Non-Ferrous Metals for Chemical Analysis.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water [see IS : 1070-1960 Specification for Water, Distilled Quality (*Revised*)] shall be employed in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

• Second revision issued in 1967.

4. DETERMINATION OF COPPER BY THE ELECTROLYTIC METHOD

4.1 Outline of the Method-The sample is dissolved in sulphuric acid-nitric acid mixture and copper deposited electrolytically from the solution. In case of copper, having a purity of less than 99.5 percent, impurities are removed by ferric hydroxide and then copper deposited electrolytically.

4.2 Apparatus -The following platinum electrodes (see Fig. 1) are recommended, but strict adherence to the shape and size of the electrodes is not essential. For agitation of electrolyte in order to decrease the time of deposition, one of the types of rotating forms of electrodes, generally available, may be employed.

4.2.1 Cathode-It may be formed either from plain or perforated sheet or from wire gauze.

4.2.1.1 Gauze cathodes preferably made from gauze containing 400 mesh/cm* should be used. The wire used for making gauze should be approximately 0.20 mm in diameter. Cathode should be stiffened by doubling the gauze for about 3 mm on the top and the bottom or by reinforcing the gauze at the top and bottom with platinum ring or band.

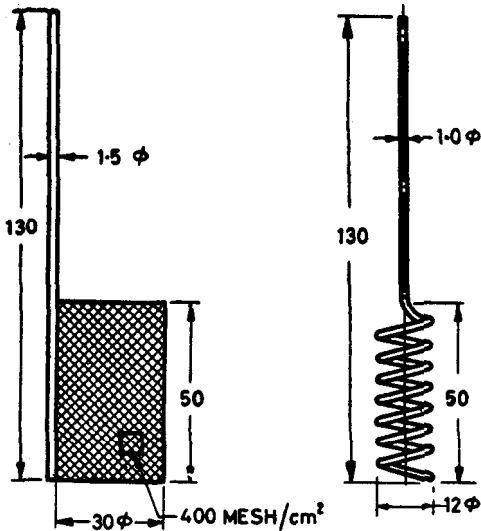
4.2.1.2 The diameter of the cylinder should be approximately 30 mm and the height 50 mm. The stem should be made from platinum alloy wire, such as platinum-iridium, platinum-rhodium or platinum-ruthenium having diameter of approximately 1.5 mm. It should be flattened and welded along the entire length of the gauze. The overall height of the cathode should be approximately 130 mm.

4.2.2 Anode-Either a spiral or a gauze anode should be used. The spiral of seven turns with a height of approximately 50 mm and diameter of 12 mm should be made from 1.0 mm or larger platinum wire, the overall height being 130 mm. The gauze anode should be made of the same material and of the same general design as platinum gauze cathode mentioned under 4.2.1. The diameter of the cylinder should be approximately 12 mm and the height 50 mm, the overall height of the anode being 130 mm.

4.3 Reagents

4.3.1 Sulphuric Acid-Nitric Acid Mixture — Add slowly, with stirring, 300 ml of concentrated sulphuric acid [sp gr 1.84; conforming to IS : 266-1961 Specification for Sulphuric Acid (Revised)] to 750 ml of water, cool and add 210 ml of concentrated nitric acid (see 4.3.8).

4.3.2 Urea -solid.

1A Cylindrical Platinum
Cathode1B Spiral Platinum
Anode

All dimensions in millimetres.

FIG. 1 CYLINDRICAL PLATINUM CATHODE AND
SPIRAL PLATINUM ANODE

4.33 Ethanol or Methanol -95 percent (v/v).

4.34 Ferric Nitrate Solution — Dissolve 70 g of ferric nitrate crystals [$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$] in water and dilute to one litre.

4.35 Concentrated Ammonium Hydroxide — 20 percent.

4.36 Ammonium Sulphate Solution — 1:99 (w/v).

4.3.7 Dilute Sulphuric Acid- 1 : 1 and 1 : 2 (v/v).

4.3.8 -Concentrated Nitric Acid — sp gr 1.42 (conforming to *IS : 264-1950 Specification for Nitric Acid).

4.4 Procedure (for Copper Having a Purity of 99.5 percent and Over)

4.4.1 Transfer 5 g of the accurately weighed sample into a 250-ml beaker provided with a cover glass and add 42 ml of sulphuric acid-nitric acid mixture. After the initial reaction is over, heat to 80° to 90°C until completely dissolved and brown fumes are expelled.

*Since revised .

4.4.2 Wash down the cover and sides of the beaker and dilute the solution sufficiently to cover the cathode cylinder. Insert the electrodes, the cathode having been accurately weighed; ~~cover with a pair of split cover glass~~ and electrolyze for 16 hours at a current density of 0.6 A/dm². When the solution becomes colourless reduce the current density to 0.3 A/dm²; add one gram of urea and wash down the cover glasses and sides of the beaker and continue electrolysis until the deposition of copper is complete as indicated by absence of plating on the new surface of the electrode obtained by the raised level of the solution.

4.4.3 Without interrupting the current, syphon off the electrolyte, at the same time adding water to maintain the level of the solution. Remove the cathode quickly, while washing further with water, rinse it with water in a beaker and then dip it in two successive baths of ethanol or methanol. Dry the cathode in an air oven at 110°C for three to five minutes, cool and weigh for copper.

4.4.4 Calculation

$$\text{Copper, percent} = \frac{A}{B} \times 100$$

where

A = weight in g of copper, and

B = weight in g of the sample taken.

4.5 Procedure (for Copper Having a Purity of Less Than 99.5 percent)

4.5.1 Dissolve the sample in the acid mixture as under 4.4.1. Evaporate until all the nitric acid is expelled. Dissolve the residue in 70 ml of water, add 3 ml of ferric nitrate solution, heat just to boiling and precipitate the iron by adding concentrated ammonium hydroxide in slight excess keeping all the copper in solution. Filter and wash the precipitate with ammonium sulphate solution and preserve the filtrate and the washings.

4.5.2 Wash the precipitate back into the beaker and dissolve in dilute sulphuric acid (1 : 1). Re-precipitate with concentrated ammonium hydroxide keeping the volume as little as possible. Filter through the same paper and wash with ammonium sulphate solution. Again wash the precipitate back into the beaker, dissolve, re-precipitate, filter and wash. Combine all the filtrates and evaporate to get the proper volume for electrolysis. Acidify with dilute sulphuric acid (1 : 2) and add 15 ml in excess, and then add 2 ml of concentrated nitric acid. Complete the determination as described under 4.4.2 to 4.4.4.

5. DETERMINATION OF ARSENIC AND ANTIMONY BY THE CO-PRECIPIATION (IODIMETRIC) METHOD

5.1 Outline of the Method -The sample is dissolved in nitric acid and hydrochloric acid, Arsenic and antimony are co-precipitated with ferric hydroxide, dissolved in sulphuric acid and determined, after reduction and distillation as their trichlorides, volumetrically, by titration with standard iodine solution.

5.2 Apparatus — An all glass distillation apparatus as shown in Fig. 2 shall be used.

5.3 Reagents

5.3.1 *Concentrated Nitric Acid* -see 43.8.

5.3.2 *Concentrated Hydrochloric Acid* — sp gr 1.16 [conforming to IS : 265-1962 Specification for Hydrochloric Acid (Revised)].

5.3.3 *Ferric Nitrate Solution* — Dissolve 20 g of ferric nitrate crystals [$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$] in water and dilute to 100 ml.

5.3.4 *Concentrated Ammonium Hydroxide* — 20 percent.

5.3.5 *Dilute Ammonium Hydroxide* — 1 : 4 (v/v).

5.3.6 *Dilute Sulphuric Acid* — 1 : 3 (v/v).

5.3.7 *Carbon Dioxide* — gas.

5.3.8 *Hypophosphorus Acid* — 50 percent (v/v).

5.3.9 *Sodium Sulphite* — solid.

5.3.10 *Sodium Hydroxide Solution* — 20 percent (w/v).

5.3.11 *Methyl Red Indicator Solution* — Dissolve 0.1 g of the reagent in 60 ml of rectified spirit and make up to 100 ml with water.

5.3.12 *Dilute Hydrochloric Acid* — 1 : 1 (v/v).

5.3.13 *Sodium Bicarbonate* — solid.

5.3.14 *Starch Solution* — Make a suspension of one gram of soluble starch in about 10 ml of water and add it carefully to 100 ml of boiling water. Boil for two or three minutes and cool. Prepare the solution fresh as needed.

5.3.15 *Potassium Iodide Solution* — Dissolve 100 g of potassium iodide in water and dilute to one litre.

5.3.16 *Standard Iodine Solution* — 0.02 N approx. Dissolve 254 g of iodine and 8 g of potassium iodide in 25 ml of water. When solution is

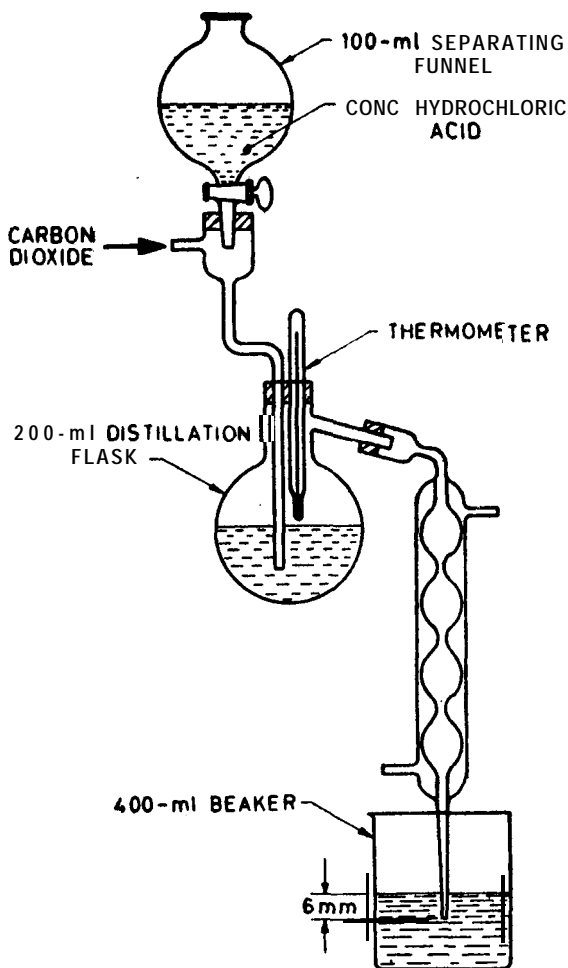


FIG. 2 APPARATUS FOR DETERMINATION OF ARSENIC AND ANTIMONY BY DISTILLATION

complete, dilute to one litre. Store in a cool place in a dark-coloured glass-stoppered bottle. Standardize against pure arsenic trioxide (As_2O_3).

5.3.17 Tartaric Acid Solution — 25 percent (w/v).

5.4 Procedure

5.4.1 Transfer 20 g or less of the accurately weighed sample containing not more than 0.005 g of arsenic to a 600-ml beaker and add 50 ml of concentrated nitric acid and 20 ml of concentrated hydrochloric acid. When the solution is complete, add 10 ml of ferric nitrate solution and boil for one to two minutes. Dilute to 400 ml and add concentrated ammonium hydroxide until the acid is neutralized and the smell of ammonia persists. Boil for five minutes and allow the precipitate to settle. Filter through a rapid filter paper and wash twice with hot dilute ammonium hydroxide solution, leaving some copper with the precipitate. Dissolve the precipitate through the filter paper with 25 ml of hot dilute sulphuric acid and wash thoroughly with hot water, catching the solution in the original beaker.

5.4.2 Transfer the clear solution to the distillation flask and fit up the apparatus as shown in Fig. 2. Evaporate to white fumes while passing a current of carbon dioxide through the apparatus. Allow the solution to cool, place a 400-ml beaker containing 50 ml of ice-cold water under the condenser so that the tip of the condenser is dipped about 6 mm in water. Add 50 ml of concentrated hydrochloric acid and one millilitre of hypophosphorus acid to the distillation flask.

5.4.3 Place about 80 ml of concentrated hydrochloric acid in the 100-ml separating funnel and pass a stream of carbon dioxide through the apparatus. Heat the solution in the distillation flask while dropping concentrated hydrochloric acid from the separating funnel at a rate that the temperature of the solution is maintained at about 110°C. Continue distillation until all the acid from the separating funnel is added: without disturbing the current of carbon dioxide, remove the receiver and rinse the end of the condenser with water. Remove the beaker and preserve for the determination of arsenic (see 5.4.5).

5.4.4 Place a 250-ml beaker containing 50 ml of water under the condenser so that the tip of the condenser dips under water. Add 80 ml of concentrated hydrochloric acid in the funnel and pour it gradually into the flask maintaining the temperature of the solution at about 150°C until all the acid has been added. Remove the beaker containing the distillate, add one gram of sodium sulphite, place cover on the beaker and boil until the volume is reduced to 30 ml. Reserve the solution for the determination of antimony (see 5.4.6).

5.4.5 Cool the distillate in the beaker below 30°C, neutralize with sodium hydroxide solution using methyl red as indicator and add dilute hydrochloric acid dropwise until the solution is just acidic. Cool and add 8 to 10 g of sodium bicarbonate, 5 ml of starch solution and one millilitre of potassium, iodide solution. Titrate with Standard iodine solution to a persistent blue tint.

5.4.6 To the distillate reserved under 5.4.4, for the determination of antimony, add 80 ml of water and 10 ml of tartaric acid solution, neutralize with sodium hydroxide to methyl red and make just acidic with dilute hydrochloric acid. Cool, add 10 g of sodium bicarbonate, 5 ml of starch solution, one millilitre of potassium iodide and titrate with standard iodine solution.

5.4.7 Carry out a blank determination following the same procedure and using the same amounts of all reagents but without the sample.

5.5 Calculations

$$\text{Arsenic, percent} = \frac{(A_1 - B_1) C \times 3.75}{D}$$

$$\text{Antimony, percent} = \frac{(A_2 - B_2) C \times 6.09}{D}$$

where

A_1 = volume in ml of the standard iodine solution required for the sample,

B_1 = volume in ml of the standard iodine solution required for the blank,

c = normality of the standard iodine solution,

D = weight in g of the sample taken,

A_2 = volume in ml of the standard iodine solution required for the sample, and

B_2 = volume in ml of the standard iodine solution required for the blank.

6. DETERMINATION OF ARSENIC BY THE HYPOPHOSPHITE IODIMETRIC (ALTERNATE) METHOD (APPLICABLE IN ABSENCE OF Se AND Te)

6.1 Outline of the Method -Arsenic in the solution of the sample is reduced by sodium hypophosphite to metallic arsenic in acid medium, and the metal oxidized by a measured excess of standard iodine solution to pentavalent arsenic. The excess of iodine is back-titrated with standard sodium arsenite solution.

6.2 Reagents

6.2.1 Dilute Sulphuric Acid- 1: 3 (v/v).

6.2.2 Concentrated Nitric Acid-see 4.3.8.

6.2.3 Concentrated Hydrochloric Acid — see **5.3.2.**

6.2.4 Sodium Hypophosphite — solid.

6.2.5 Dilute Hydrochloric Acid — 1 : 3 (v/v).

6.3.6 Ammonium Chloride Solution — 5 percent (w/v).

6.2.7 Standard Iodine Solution (1 ml = 0.00015 g of As) — 0.01 N. Dissolve 1.27 g of re-sublimed iodine and 4 g of potassium iodide in 25 ml of water. When the solution is complete, dilute to one litre with water and store in a dark-coloured glass-stoppered bottle. Standardize against standard arsenious oxide solution.

6.2.8 Sodium Carbonate — solid.

6.2.9 Starch Solution -see **5.3.14.**

6.2.10 Standard Sodium Arsenite Solution-001 N. Dissolve **0.495** g of arsenious oxide in about 4 ml of sodium hydroxide solution (10 per cent), and dilute to about 200 ml. Add dilute hydrochloric acid (1 : 20) until just acidic. Add about 2 g of sodium bicarbonate and dilute to one litre in a volumetric flask.

6.3 Procedure

6.3.1 Transfer **5** g of the accurately weighed sample to a beaker and add 80 ml of dilute sulphuric acid and 30 ml of concentrated nitric acid. Evaporate the solution to fumes until all nitric acid is expelled. Cool, dilute with 75 ml of hot water and boil gently to dissolve the salts. Transfer the solution to a **500-ml** flask and rinse the beaker with 100 ml of concentrated hydrochloric acid. Add 2 g of sodium hypophosphite, warm to brisk effervescence avoiding boiling and further add 2 g of the hypophosphite until no further effervescence occurs. Finally add 12 g of hypophosphite and boil under reflux for 15 minutes.

6.3.2 Filter **off the** precipitated arsenic immediately, while hot, through a paper-pulp pad and wash with 100 ml of dilute hydrochloric acid containing 3 g of hypophosphite, and then about seven times with ammonium chloride solution. Discard the filtrate and transfer the residue immediately to a **800-ml** tall-type beaker, rinsing with about 50 ml of water.

6.3.3 Add a measured excess of the standard iodine solution, stir, allow to stand for five minutes, then dilute if necessary. Add about 2 g of sodium carbonate followed by a few millilitres of starch solution. Titrate with standard sodium arsenite solution to a pronounced lightening of the **colour**. Discharge the coloration by adding 3 ml more of the standard sodium arsenite solution. Wash continuously with vigorous shaking and back-titrate with standard iodine solution.

6.3.4 Carry out a blank determination by following the same procedure and using the same amounts of all reagents but without the sample.

6.4 Calculation

$$\text{Arsenic, percent} = \frac{[A - (B + C)] \times 0.015}{D}$$

where

A = volume in ml of the standard iodine solution,

B = volume in ml of the standard iodine solution equivalent to standard sodium arsenite solution,

C = volume in ml of the standard iodine solution required for the blank, and

D = weight in g of the sample taken.

7. DETERMINATION OF BISMUTH BY THE IODIDE (PHOTOMETRIC) METHOD

7.1 **Outline of the Method**-Bismuth is collected and separated from copper by precipitation with ammonia in the presence of ferric iron and is determined photometrically using potassium iodide as complexing agent. The photometric measurement is made at approximately 440 m μ .

7.2 Reagents

7.2.1 Concentrated Nitric Acid- see 4.3.8.

7.2.2 Ferrous Ammonium Sulphate — solid.

7.2.3 Concentrated Ammonium Hydroxide — 20 percent.

7.2.4 Ammonium Carbonate Solution-Dissolve 100 g of ammonium carbonate in water and dilute to one litre.

7.2.5 Dilute Ammonium Hydroxide — 1:9 (v/v).

7.2.6 Dilute Sulphuric Acid- 1:4 (v/v).

7.2.7 Potassium Iodide Solution — 10 percent (w/v).

7.2.8 Sodium Hypophosphite Solution — 25 percent (w/v).

7.2.9 Phosphoric Acid- 85 percent.

7.2.10 Standard Bismuth Solution (1 ml = 0.0001 g of Bi) — Dissolve 0.1000 g of pure bismuth metal in 10 ml of dilute nitric acid (1 : 1), and 2 ml of concentrated sulphuric acid and evaporate to fumes, Add few

drops of concentrated nitric acid. Add 20 ml of water, heat to boiling and dilute to one litre.

7.3 Procedure

73.1 Transfer 10 to 50 g (depending upon bismuth content) of the accurately weighed sample to a 400-ml beaker and add concentrated nitric acid at the rate of 40 ml per 10 g of the sample. When the dissolution is complete, add 0.25 g of ferrous ammonium sulphate and boil for two minutes. Dilute to 200 ml with water and add concentrated ammonium hydroxide until the precipitated copper hydroxide is redissolved, and then add a little in excess. Add 10 ml of ammonium carbonate solution and heat to boiling. Allow the solution to stand overnight. Filter through a rapid filter paper and wash with hot dilute ammonium hydroxide. Dissolve the precipitate through the filter paper with 20 ml of hot dilute sulphuric acid and wash with hot water. Reprecipitate with concentrated ammonium hydroxide, boil for two minutes and allow the precipitate to settle overnight. Filter and wash with hot dilute ammonium hydroxide solution to remove the copper completely and then twice with hot water. Dissolve the precipitate as before.

73.2 Neutralize the solution with dilute ammonium hydroxide and then acidify with dilute sulphuric acid avoiding excess of the acid. Add 4 ml of sodium hypophosphite solution, 10 ml of potassium iodide solution, few millilitres of phosphoric acid, and make the volume to 100 ml. Allow the solution to stand for 10 minutes.

73.3 Transfer a suitable volume of the solution to an absorption cell and take the photometric reading using a light filter at 440 m μ .

73.4 *Calibration Curve*— Take a series of standard bismuth solutions and the reagent blank. Using the same quantities of the reagents, carry through the procedure as described under 7.3.2 and 7.3.3 and record the photometric readings of the standard solutions along with the blank. Correct for the blank and draw a calibration curve by plotting the photometric reading of the solutions against milligrams of bismuth per 100 ml of the solution.

7.4 *Calculation*—Convert the photometric reading of the sample solution to milligrams of bismuth by means of a calibration curve and calculate the percentage of bismuth as follows:

$$\text{Bismuth, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = weight in mg of the bismuth found in the aliquot of the sample, and

B = weight in g of the sample represented by the aliquot taken.

8. DETERMINATION OF IRON

8.1 Outline of the Method—Iron in the solution of the sample is reduced with stannous chloride, excess of which is destroyed by mercuric chloride. The reduced iron is then titrated with standard potassium dichromate solution using sodium diphenylamine sulphonate as indicator. Alternatively, iron is determined photometrically with thiocyanate solution and measuring the absorbance at approximately 480 m μ .

8.2 Reagents

8.2.1 Dilute Nitric Acid—1:1 and 1:99 (v/v).

8.2.2 Concentrated Nitric Acid—see 4.3.8.

8.2.3 Perchloric Acid—70 percent.

8.2.4 Hydrobromic Acid—48 percent.

8.2.5 Concentrated Ammonium Hydroxide—20 percent.

8.2.6 Ammonium Chloride Solution—20 g per litre.

8.2.7 Dilute Hydrochloric Acid—1:1 (v/v).

8.2.8 Stannous Chloride Solution—Dissolve, by heating, 60 g of pure stannous chloride in a mixture of 400 ml of concentrated hydrochloric acid and 600 ml of water until the solution is complete. Cool, add a few pieces of granulated tin and preserve the solution in an air-tight amber-coloured bottle to prevent oxidation.

8.2.9 Mercuric Chloride Solution—saturated.

8.2.10 Sulphuric Acid-Phosphoric Acid Mixture—Add slowly 150 ml of concentrated sulphuric acid to 700 ml of water with continuous stirring. Add to this 150 ml of syrupy phosphoric acid. Destroy any oxidizable impurities by adding potassium permanganate solution (0.1 N) drop by drop until the pink colour of permanganate persists, and cool the solution.

8.2.11 Sodium Diphenylamine Sulphonate Indicator Solution—Dissolve 0.32 g of barium diphenylamine sulphonate in 100 ml of hot water. Add 0.5 g of sodium sulphate, stir, and filter off the precipitate of barium sulphate. Store in a dark-coloured bottle.

8.2.12 Standard Potassium Dichromate Solution—Dry pure potassium dichromate in an oven at 105° to 110°C for one and a half hours. Cool in a desiccator and transfer exactly 0.4904 g to a one-litre volumetric

flask. Dissolve the salt in water and make up the volume to the mark. Mix well.

8.2.13 *Ammonium Thiocyanate Solution* -Dissolve 10 g of ammonium thiocyanate in water and dilute to one litre.

8.2.14 *Standard Iron Solution (1 ml = 0.05 mg of Fe)* -Dissolve **0.351** g of ferric ammonium sulphate [Fe (NH₄)₂ (SO₄)₃ · 6 H₂O] in 5 ml of dilute nitric acid (1 : 1). Dilute to one litre in a volumetric flask and mix.

8.3 Procedure [By the **Dichromate**(Volumetric) Method]

8.3.1 Transfer **5 g** of the accurately weighed sample, from which extraneous iron has been removed, to a beaker. Add 50 ml of dilute nitric acid (1 : 1) and heat to expel brown fumes. Dilute the solution to 100 ml with hot water and digest metastannic, if any, on a steam bath. Filter through a close-textured paper into a **400-ml** beaker. Wash with dilute nitric acid (1 : 99). Preserve the filtrate.

8.3.2 Transfer the paper and contents to the original beaker. Add 15 ml of concentrated nitric acid and 10 ml of perchloric acid. Heat gently until white fumes appear. Cool and wash down the sides of the beaker. Cover and add **10 ml** of hydrobromic acid. Remove the cover and boil gently until white fumes appear. Repeat the hydrobromic acid treatment to expel any traces of tin and antimony. Add **15 to 20 ml** of water to dissolve these soluble salts. Combine the solution with the filtrate preserved under 8.3.1.

8.3.3 Dilute the solution to 150 ml. Add concentrated ammonium hydroxide solution until slightly but distinctly alkaline. Boil gently for a few minutes. Allow the precipitate to settle for two minutes, filter while hot. Wash the precipitate a few times alternately with ammonium chloride solution and hot water to remove traces of the soluble salts.

8.3.4 Dissolve the precipitate on the filter paper with 20 ml of dilute hydrochloric acid and wash thoroughly with hot water to remove the last traces of iron. Collect the solution and the washings in **the** beaker in which precipitation was done. Reprecipitate iron and dissolve it as before.

8.3.5 Concentrate the final solution to about 20 ml and heat to boiling. To the boiling solution, add stannous chloride solution **drop-**wise with continuous stirring until the solution becomes **colourless**. Add one drop of stannous chloride in excess. Dilute the solution to 50 ml and cool to *room* temperature. Add 10 ml of mercuric chloride solution in one instalment. At this stage, silky white precipitate appears; if it fails to appear or if a black precipitate appears, reject the test. If the precipitate is too much, add further 5 ml of mercuric chloride solution. **Keep** for about 2 to 5 minutes and add 15 ml of sulphuric acid-phosphoric acid

mixture and dilute to 100 ml. Add 3 to 4 drops of sodium diphenylamine sulphonate indicator solution and titrate with standard dichromate solution while stirring continuously until the green colour begins to darken. Continue titration until a drop of dichromate solution produces a suitable violet blue coloration.

8.3.6 Carry out a blank determination following the same procedure and using the same amounts of all the reagents, but without the sample.

8.3.7 Calculation

$$\text{Iron, percent} = \frac{(A - B) C \times 5.59}{D}$$

where

A = volume in ml of the standard dichromate solution required to titrate the sample solution,

B = volume in ml of the standard dichromate solution required to titrate the blank,

C = normality of the standard dichromate solution, and

D = weight in g of the sample taken.

8.4 Procedure [By the Ammonium Thiocyanate (Photometric) Method]

8.4.1 Weigh accurately about 10 g of the sample into a 600-ml tall-type lipless beaker covered with a well-fitting cover glass and dissolve by adding 100 ml of dilute nitric acid (1 : 1). Allow to stand on a hot plate until all nitrous fumes are expelled. Dilute to approximately 300 ml with warm water and add concentrated ammonium hydroxide until all basic salts have been dissolved. Bring to the boil carefully, place on a hot plate and allow to stand for about one hour to affect coagulation of iron hydroxide. Filter and wash several times with warm dilute ammonium hydroxide.

8.4.2 Dissolve the precipitate back into the original beaker with 10 ml of hot dilute hydrochloric acid, wash thoroughly with hot water, dilute to approximately 300 ml with warm water. Add concentrated ammonium hydroxide in slight excess to precipitate iron hydroxide, bring to the boil carefully and place the beaker on a hot plate to allow the precipitate to settle. Filter and wash well with hot water. Add 15 ml of hot dilute hydrochloric acid to the same beaker in which the precipitate was made to dissolve the adhering iron hydroxide. Pour the hot acid from beaker through the filter paper into a 150-ml beaker and wash the beaker and filter paper carefully with hot distilled water. Cool the solution, transfer to a 100-ml graduated flask and make up to the mark with water.

8.4.3 Transfer a suitable aliquot containing less than 1.0 mg of iron to a 200-ml volumetric flask. Add sufficient dilute nitric acid (1 : 1) so that its total content in the solution is 10 ml. Add one millilitre of hydrogen peroxide and mix. Add 40 ml of ammonium thiocyanate solution, dilute to the mark and mix *thoroughly.

8.4.4 Transfer a suitable portion of the solution to an absorption cell and measure the absorbance at approximately 486 m μ .

8.4.5 *Calibration Curve* — Take a series of standard iron solutions and the reagent blank. Using the same quantities of the reagents, follow the procedure as under 8.4.3 and 8.4.4 and record the photometric readings of the standard solutions along with the blank. Correct for the blank and draw a calibration curve by plotting photometric readings of the solutions against milligrams of iron per 100 ml of the solution.

8.4.6 *Calculation -Convert* the photometric reading of the sample solution to milligrams of iron by means of calibration curve and calculate the percentage of iron as follows:

$$\text{Iron, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = weight in mg of iron found in the aliquot solution used,
and

B = weight in g of the sample represented by the aliquot taken.

9. DETERMINATION OF LEAD **BY** THE COLORIMETRIC METHOD

9.1 Outline of the Method — Lead is deposited electrolytically from nitric acid solution of the sample and estimated **colorimetrically** as sulphide.

9.2 Apparatus — same as under 4.2.

9.3 Reagents

9.3.1 Dilute Nitric Acid — 1 : 1 (v/v).

9.3.2 Hydrogen Peroxide Solution — 3 percent.

9.3.3 Acidified Hydrogen Sulphide Solution — Saturate dilute sulphuric acid (1 : 99) with hydrogen sulphide gas.

9.3.4 Ammonium Acetate Solution — Dissolve 23 g of ammonium acetate in water and 3 ml of glacial acetic acid and dilute to 100 ml (approximately 3 N).

*The colour is stable for 30 minutes, but should be protected from direct sunlight.

IS : 440 - 1964

9.35 Sucrose Solution — 50 percent (w/v).

93.6 Standard Lead Acetate Solution (1 ml = 0.0001 g of Pb) — Dissolve 0.1831 g of lead acetate [$\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$] in water containing a little acetic acid, and dilute to one litre.

9.4 Procedure

9.4.1 Transfer 10 g of the accurately weighed sample into a 400-ml beaker, cover and dissolve in 60 ml of dilute nitric acid. When dissolution is complete boil gently to expel brown fumes. Wash down the cover glass and the sides of the beaker and dilute to 250 ml.

9.4.2 Insert the electrodes into the solution, cover with a pair of split glass and electrolyze for two hours at a current density of 1.25 to 1.50 A/dm² with agitation of the electrolyte or run the electrolysis overnight at a current density of 0.5 A/dm². Wash down the cover glass and sides of the beaker. When the electrolysis is complete as indicated by absence of darkening of the fresh anode surface, syphon off the electrolyte, at the same time filling the beaker with water. Remove the anode quickly and rinse thoroughly with water.

9.4.3 Dissolve the deposit of lead peroxide in 5 ml of dilute nitric acid and few drops of hydrogen peroxide solution. Evaporate to dryness and take up with water. Transfer to a 100-ml volumetric flask and dilute to the mark and mix well.

9.4.4 Pipette out a suitable aliquot into a 100-ml Nessler tube. Add 6 ml of ammonium acetate solution, 3 drops of dilute nitric acid, one millilitre of sucrose solution and 6 ml of hydrogen sulphide solution. Make up the volume to 100 ml with water. Mix well and compare the colour against those of series of solutions each containing measured volume of the standard lead solution to which all the reagents have been added in the same quantities as in the test solution. It is important to add hydrogen sulphide solution simultaneously to the standard and the test solutions.

9.5 Calculation

$$\text{Lead, percent} = \frac{A}{B} \times 0.01$$

where

A = volume in ml of the standard lead solution required for matching the colour of the test solution, and

B = weight in g of the sample represented by the aliquot taken.

10. DETERMINATION OF NICKEL BY THE DIMETHYLGLYOXIME (GRAVIMETRIC) METHOD

10.1 Outline of the Method — After the removal of copper, nickel is precipitated with dimethylglyoxime in alkaline medium, dried and weighed.

10.2 Reagents

10.2.1 Mixed Acids — Pour 240 ml of concentrated sulphuric acid (sp gr 1.84) into 600 ml of water, cool and add 160 ml of concentrated nitric acid (sp gr 1.42).

10.2.2 Tartatic Acid Solution-Dissolve 250 g of tartaric acid in water and dilute to one litre.

10.2.3 Concentrated Ammonium Hydroxide — 20 percent.

10.2.4 Dimethylglyoxime Solution — Dissolve 10 g of dimethylglyoxime in one litre of rectified spirit, 95 percent (v/v) [conforming to IS : 323-1959 Specification for Rectified Spirit (Revised)]. Filter before use.

10.3 Procedure-Weigh 5 g of the sample accurately and dissolve in 50 ml of mixed acids, cool if necessary. When action has almost ceased, warm to complete solution and to remove all nitrous fumes. Cool and dilute to approximately 300 ml and remove the copper by electrolysis as under 4.4.2. Add 10 ml of tartaric acid solution. Neutralize with concentrated ammonium hydroxide and add one millilitre in excess. The solution should remain clear after this treatment. Heat nearly to boiling and add with stirring 0.4 ml of dimethylglyoxime solution for each one milligram of nickel present, then add 5 to 10 ml of the dimethylglyoxime solution in excess, pouring directly into the solution and not down the beaker wall. Stir the mixture vigorously and allow to cool to room temperature while stirring occasionally. Filter through a sintered glass crucible and wash well with hot water. Dry the precipitate at 110° to 120°C to constant weight and weigh as nickel dimethylglyoxime.

10.4 Calculation

$$\text{Nickel, percent} = \frac{A}{B} \times 2032$$

where

A = weight in g of nickel dimethylglyoxime, and

B = weight in g of the sample taken.

11. DETERMINATION OF SELENIUM BY THE GRAVIMETRIC METHOD

11.1 Outline of the Method — Selenium and tellurium are separated from copper by precipitation with ammonium hydroxide in presence of ferric

IS : 440 - 1964

iron. The precipitate is dissolved in hydrochloric acid and selenium separated by sulphur dioxide, leaving tellurium in the filtrate.

11.2 Reagents

11.2.1 *Concentrated Nitric Acid* — see 4.3.8.

11.2.3 *Ferric Nitrate Solution* — see 4.3.4.

11.2.3 *Concentrated Hydrochloric Acid* — see 5.3.2.

11.3.4 *Concentrated Ammonium Hydroxide* — 20 percent.

11.2.5 *Dilute Ammonium Hydroxide* — 1 : 9 (v/v).

11.2.6 *Dilute Hydrochloric Acid* — 1 : 1 (v/v).

11.2.7 *Sulphur Dioxide Solution* — Pass sulphur dioxide gas in water until saturated.

11.2.8 *Rectified Spirit* — 95 percent (v/v) [conforming to IS : 323-1959 Specification for Rectified Spirit (Revised)].

11.3 Procedure

11.3.1 Take about 50 g of the accurately weighed sample in a large beaker and dissolve in concentrated nitric acid avoiding excess as far as possible. Add 5 to 10 ml of the ferric nitrate solution and evaporate to expel most of the free acid. Dilute to 500 ml and treat the solution with excess of concentrated ammonium hydroxide. Boil the solution for a minute or two and allow to settle overnight. Filter off the ferric hydroxide precipitate and wash with dilute ammonium hydroxide.

11.3.2 Dissolve the precipitate on the filter paper in hot dilute hydrochloric acid and wash thoroughly with hot water. Add 200 ml of concentrated hydrochloric acid and cool to 20°C. Add 20 ml of sulphur dioxide solution and allow to stand for 30 minutes. Filter through a weighed sintered glass crucible (porosity 4), wash with dilute hydrochloric acid and then with cold water and rectified spirit. Preserve the filtrate for tellurium determination (see 12). Dry the precipitate at 105 to 110°C to constant weight and weigh as selenium metal.

11.4 Calculation

$$\text{Selenium, percent} = \frac{A}{B} \times 100$$

where

A = weight in g of selenium, and

B = weight in g of the sample taken.

12. DETERMINATION OF TELLURIUM BY THE GRAVIMETRIC METHOD

12.1 Outline of the Method — Tellurium is determined from the filtrate from selenium determination, after dilution and adjustment of the acidity of the solution and precipitation with sulphur dioxide.

12.2 Reagents

12.2.1 *Tartaric Acid* — solid.

12.2.2 *Concentrated Ammonium Hydroxide* — 20 percent.

12.2.3 *Sulphur Dioxide Solution* — saturated solution in water.

12.2.4 *Hydrarine Hydrochloride Solution* — Dissolve 15 g of hydrazine hydrochloride in water and dilute to 100 ml.

12.2.5 *Rectified Spirit* — see 11.2.8.

12.2.6 *Carbon Dioxide* — gas.

12.3 Procedure -To the filtrate preserved under 11.2.2, add 2 g of tartaric acid; dilute to 600 ml with water and add 50 ml of concentrated ammonium hydroxide. Add 20 ml of sulphur dioxide solution and then 10 ml of hydrazine hydrochloride solution. Heat to boiling and boil for two minutes. Add 10 ml more of sulphur dioxide solution and allow to settle overnight. Filter through a weighed sintered glass crucible (porosity 4); wash with hot water and finally with rectified spirit. Dry to constant weight at 105 to 110°C to constant weight in an atmosphere of carbon dioxide and weigh as tellurium metal.

12.4 Calculation

$$\text{Tellurium, percent} = \frac{A}{B} \times 100$$

where

A = weight in g of tellurium, and

B = weight in g of the sample taken for the test under 11.3.1.

13. DETERMINATION OF OXYGEN BY THE COMBUSTION METHOD

13.1 Outline of the Method — Oxygen is determined from the weight of water formed on combustion in a current of hydrogen.

13.2 Apparatus -The type of apparatus to be used is shown in Fig. 3.

13.2.1 Furnace-The furnace consists of a horizontal combustion tube heated by either electricity or gas. A combustion tube, 600 mm long and

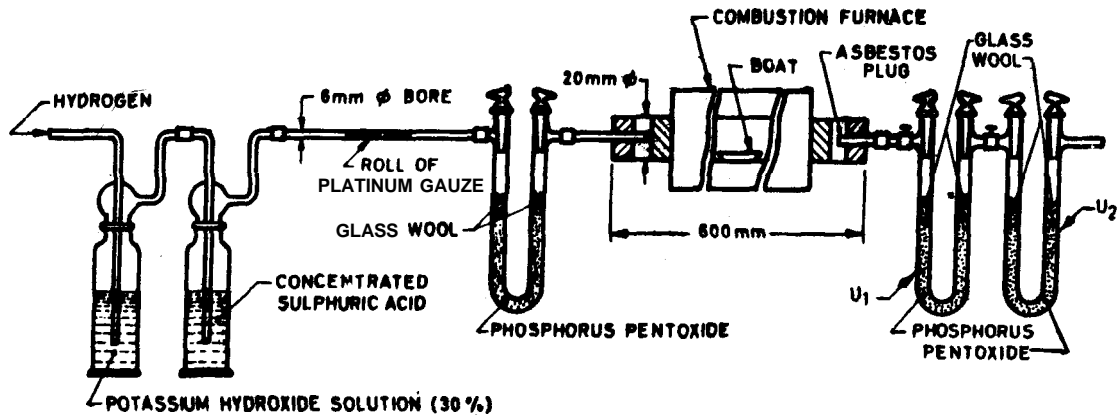


FIG. 3 APPARATUS FOR DETERMINATION OF OXYGEN BY COMBUSTION METHOD

20 mm bore, made either of fused silica, quartz, or porcelain, glazed on one or both sides, shall be used.

13.2.2 Hydrogen -Hydrogen may be obtained from hydrogen cylinder or generated by the action of hydrochloric acid on zinc in Kipp's apparatus.

13.2.3 Hydrogen Purifier — Hydrogen shall be free from oxygen and moisture. The gas is, therefore, first passed through wash bottles containing potassium hydroxide and sulphuric acid, and then through a silica tube of 6 mm bore containing platinum gauze, and maintained at a red heat whereby water is formed if any oxygen is present. The water formed is absorbed by passing through a U-tube containing phosphorus pentoxide.

13.2.4 Absorption Tubes -The product of combustion, water, is absorbed in a tared absorption tube containing phosphorus pentoxide to which is connected a guard tube containing phosphorus pentoxide. The phosphorus pentoxide tubes should be charged with alternate layers of phosphorus pentoxide and glass wool, beginning and ending with the latter. Phosphorus pentoxide, on absorbing water, tends to clog up the absorption tube. To avoid this, and also to increase the efficiency of the absorbent, use of small pumice stones (approximately 3 mm in diameter) shaken in phosphorus pentoxide are recommended.

13.3 Procedure — Assemble apparatus as shown in Fig. 3 and test for leaks. Pass hydrogen for 30 minutes. Weigh the tared absorption tube (U_1) after bringing the hydrogen under atmospheric pressure by momentarily opening the stopcock towards the guard tube (U_s) before disconnecting the latter. Heat to 70°C, the sample which has been drilled with considerable care to avoid overheating and cool in a desiccator. Place 50 g of the material in a large combustion boat and insert it into the combustion tube. Pass hydrogen for 30 minutes, the tube being cold. Connect the tared phosphorus pentoxide absorption tube and the guard tube. Sweep out the air by passing hydrogen for 30 minutes. Start the heating and bring the temperature to 900°C. Pass the current of hydrogen at the rate of about 100 ml per minute for 2 to 3 hours or until there is no further increase in the weight of the tared absorption tube. Cool and weigh the absorption tube as before.

13.4 Calculation

$$\text{Oxygen, percent (by weight) } = \frac{(A - B)}{C} \times 88.81$$

where

A = weight in g of the tared absorption tube after test,

B = weight in g of the tared absorption tube in blank run,
and

C = weight in g of the sample taken.

14. DETERMINATION OF TIN BY THE IODIMETRIC METHOD

14.1 **Outline of the Method** — Tin is collected and separated from copper by precipitation with ammonium hydroxide in presence of iron. After reduction of the solution, tin is determined iodimetrically.

14.2 Reagents

14.21 *Concentrated Hydrochloric Acid* — see 53.2.

14.23 *Dilute Nitric Acid* — 1:1 (v/v).

14.2.3 Ferric Nitrate Solution — see 43.4.

143.4 *Concentrated Ammonium Hydroxide* — 20 percent.

142.5 *Ammonium Chloride Solution* — one percent (v/v).

14.2.6 Dilute Hydrochloric Acid — 1:1 (v/v).

14.2.7 Antimony Chloride Solution — Dissolve 20 g of antimony chloride in water and dilute to one litre.

14.2.8 Test Lead

14.2.9 Nickel — a roll of nickel sheet.

14.2.10 Carbon Dioxide — gas.

14.2.11 *Potassium Iodide Solution* — Dissolve 100 g of potassium iodide in water and dilute to one litre.

14.2.12 Starch Solution — see 5.3.14.

14.2.13 Standard Potassium Zodate Solution — 0.05 N. Twice recrystallize potassium iodate from water and dry at 180°C to constant weight. Dissolve 1.7835 g of potassium iodate in 200 ml of water containing one gram of sodium hydroxide and add 10 g of potassium iodide. When the solution is complete, dilute to one litre in a volumetric flask. Standardize the solution against pure tin following the procedure described under **14.3**.

143 Procedure

143.1 Take, in large beakers, 100 g of the accurately weighed sample in two 50 g lots in the case of copper with copper content more than 99.5 percent, and in one lot of 50 g of the sample in the case of copper with copper content lower than 99.5 percent. For each 50 g of the

material, add 75 ml of water, 50 ml of concentrated hydrochloric acid and 150 ml of dilute nitric acid.

143.2 When the solution is complete, add 10 ml of ferric nitrate solution and boil for two minutes. Dilute to 400 ml with water and add concentrated ammonium hydroxide until the precipitated copper hydroxide is redissolved and the smell of ammonia persists. Heat to boiling and allow to settle for one hour, Filter through a rapid filter paper and wash with hot ammonium chloride solution. Dissolve the precipitate in concentrated hydrochloric acid. Combine the solutions if the sample is taken in more than one lot.

143.3 Reprecipitate with concentrated ammonium hydroxide, boil, filter and wash thoroughly with hot ammonium chloride solution. Remove the filter paper and the precipitate from the funnel and place it in a 500-ml Erlenmeyer flask. Add 200 ml of water to the flask containing the filter paper and the precipitate. Add 75 ml of concentrated hydrochloric acid, two drops of antimony chloride solution and 5 g of test lead or the roll of sheet nickel and swirl the flask to break up the filter paper and to aid in the solution of the precipitate. Stopper the flask with a three-holed rubber stopper containing an inlet tube for carbon dioxide, an air condenser, and a hole for the burette (glass plugged) (see Fig. 4). During reduction a very slow stream of carbon dioxide shall be passed through the flask. Heat the solution to boiling and boil with continuous evolution of gas for at least 45 minutes.

143.4 After reduction is complete, cool the contents of the flask to about 10°C, maintaining an atmosphere of carbon dioxide by passing carbon dioxide gas into the flask. Add 5 ml of potassium iodide solution and 5 ml of starch solution and titrate with the standard potassium iodate solution to a persistent blue colour.

143.5 Carry out a blank determination, following the same procedure and using the same quantities of all reagents but without the sample.

14.4 Calculation

$$\text{Tin, percent} = \frac{(A - B) \times C}{D} \times 100$$

where

A = volume in ml of the standard iodate solution required for the sample,

B = volume in ml of the standard iodate solution required for the blank,

C = tin equivalent of the standard potassium iodate solution in g per ml, and

D = weight in g of the sample taken.

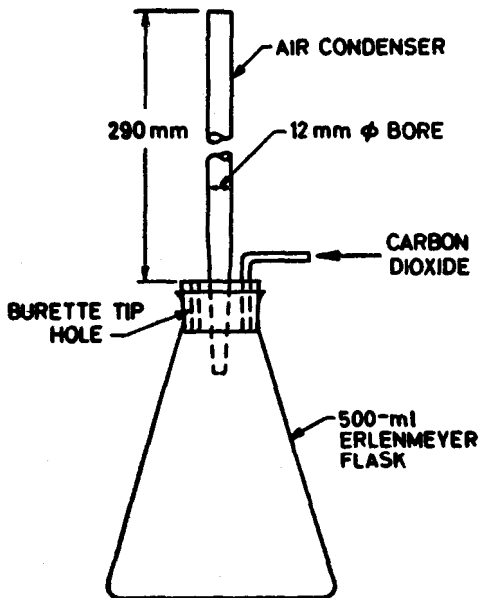


FIG. 4 APPARATUS FOR REDUCTION OF TIN

15. DETERMINATION OF PHOSPHORUS BY ALKALIMETRIC METHOD

15.1 Outline of the Method — The sample is dissolved in nitric acid and phosphorus precipitated as ammonium phosphomolybdate, filtered washed and dissolved in excess of standard alkali solution. The unreacted alkali is back-titrated against standard acid.

15.2 Reagents

15.2.1 Concentrated Nitric Acid — see 4.3.8.

15.2.2 Potassium Permanganate Solution — 10 g per litre (w/v).

15.2.3 Ammonium Nitrite Solution — 10 percent (w/v).

15.2.4 Ammonium Nitrate — solid.

15.2.5 Ammonium Molybdate Solution — Add solution (A) (see 15.2.5.1) slowly and with constant stirring to solution (B) (see 15.2.5.2) kept cool in a cold water-bath. Add 10 ml of ammonium phosphate (one gram per litre) and keep the solution at least for 24 hours. Filter the solution through filter paper No. 1 before use.

15.2.5.1 Solution (A)— Dissolve 100 g of molybdic acid (MoO_3 , 85 percent) or 118 g of ammonium molybdate in a mixture of 145 ml of concentrated ammonium hydroxide and 270 ml of water. Cool the solution.

15.2.5.2 Solution (B)— Add 490 ml of concentrated nitric acid to 1150 ml of water and cool.

15.2.6 Potassium Nitrate Wash Solution— 10 g per litre (w/v).

15.2.7 Phenolphthalein Indicator Solution— Dissolve 0.5 g of the phenolphthalein in 100 ml of 60 percent alcohol.

15.2.8 Sodium Hydroxide Solution — 0.15 N approximately.

15.2.9 Standard Hydrochloric Acid Solution — 0.15 N. Dilute 16 ml of concentrated hydrochloric acid to one litre and standardize against sodium carbonate.

15.3 Procedure

15.3.1 Transfer one to five grams of the accurately weighed sample, preferably containing 5 mg of phosphorus, to a 500-ml Erlenmeyer flask and dissolve in concentrated nitric acid using 5 ml for each gram of the sample. Heat gently to expel brown fumes and add 100 ml of water. Heat the solution and add potassium permanganate solution dropwise until a deep purple colour persists. Boil for 5 minutes, destroy the excess of potassium permanganate and manganese dioxide by adding ammonium nitrite solution dropwise until the solution is blue in colour. Boil for 5 minutes.

15.3.2 Cool the solution to 45°C and add 5 g of ammonium nitrate. Add 60 ml of ammonium molybdate solution in small portions while shaking the flask. Close the flask and shake vigorously for 5 minutes and keep it at 50 to 60°C for one hour. Filter the precipitate through a thick filter paper pulp prepared on a disc in a funnel. Wash the precipitate two to three times with dilute nitric acid, then with potassium nitrate wash solution until 10 ml of the filtrate collected in a test tube does not consume more than one drop of sodium hydroxide solution (begin testing from the third wash).

15.3.3 Transfer the filter paper and the precipitate to the flask, add 30 ml of water, stopper it and shake vigorously until the filter paper is disintegrated. Wash down the stopper, add 3 to 4 drops of phenolphthalein indicator solution and while shaking the flask add from the burette standard sodium hydroxide solution until pink colour persists. Add 2 ml in excess. Dilute with about 100 ml of water and titrate with standard hydrochloric acid to the disappearance of the pink colour.

15.3.4 Carry out a blank determination following the same procedure and using the same quantity of standard sodium hydroxide solution which was added in the test, and titrate with standard hydrochloric acid using 3 to 4 drops of phenolphthalein indicator solution.

19.4 Calculation

$$\text{Phosphorus, percent} = \frac{(A - B) c \times 0.135}{D}$$

where

A = volume in ml of the standard hydrochloric acid required for the blank test,

B = volume in ml of the standard hydrochloric acid required for the sample,

C = normality of the standard hydrochloric acid, and

D = weight in g of the sample taken.

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