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IS : 1493 ( Part I ) - 1981

*Indian Standard*

METHODS OF  
CHEMICAL ANALYSIS OF IRON ORES

PART I DETERMINATION OF COMMON CONSTITUENTS

*( First Revision )*

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**BUREAU OF INDIAN STANDARDS**

MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

*Indian Standard*METHODS OF  
CHEMICAL ANALYSIS OF IRON ORES

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*( First Revision )*

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# *Indian Standard*

## METHODS OF CHEMICAL ANALYSIS OF IRON ORES

### PART I DETERMINATION OF COMMON CONSTITUENTS

### *(First Revision)*

#### 0. FOREWORD

**0.1** This Indian Standard ( First Revision ) was adopted by the Indian Standards Institution on 31 July 1981, 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 published in 1959. In this revision it was decided to cover common constituents in iron ore in Part I and other constituents in subsequent parts. A clause on calculation of correction factor for moisture has been introduced. Silver reductor method, which is a simple and rapid method, has been introduced for determination of iron content. The method for determination of sulphur has also been revised. Both these methods are based on extensive investigational work done by the committee. Another major change made in the standard relates to inclusion of provision for applying correction factor in the case of ores of hygroscopic nature by determining moisture content of the sample at the time of analysis of such ores. With the publication of this part, the methods of determination of relevant constituents prescribed in IS : 1493-1959\* will be superseded.

**0.3** In reporting the result 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†.

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#### 1. SCOPE

**1.1** This standard prescribes methods of determination of moisture, silica, iron, aluminium, phosphorus ( for  $\geq 0.05$  percent ) and sulphur in iron ores.

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\*Method of chemical analysis of iron ores.

†Rules for rounding off numerical values ( *revised* ).

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**1.2** The methods for the determination of phosphorus ( < 0.05 percent ) manganese, calcium oxide, magnesium oxide, ferrous oxide, vanadium, titanium and combined water and methods for the determination of lead, copper, zinc and arsenic which may sometimes be required will be included in subsequent parts.

## 2. SAMPLING

**2.1** Samples for the purpose of moisture determination and chemical analysis shall be drawn and prepared in accordance with IS : 1405-1966\*.

## 3. QUALITY OF REAGENTS

**3.1** Unless otherwise specified, pure chemicals shall be employed in tests and distilled water ( conforming to IS : 1070-1977† ) shall be used where the use of water as a reagent is intended.

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

## 4. GENERAL

**4.1 Use of Filter Papers** — In the methods prescribed in this standard, relative numbers of Whatman filter paper only have been prescribed, since these are commonly used. However, filter papers of any other suitable brand with equivalent porosity may be used.

**4.2** All the determinations shall be carried out in duplicate, and blank experiments shall be carried out simultaneously, especially for the determination of the trace elements like phosphorus and sulphur, for the purpose of making a corresponding correction in the results of analysis.

**4.3** Simultaneously and under the same conditions, a check analysis especially in the determination of important constituents like iron, phosphorus, etc shall be made on iron ore reference sample.

## 5. DETERMINATION OF MOISTURE IN MOISTURE SAMPLE

**5.1** Empty the entire moisture sample of 1 to 2 kg weight into a tared aluminium tray of 23 × 16 cm dimensions and 30-35 mm depth. Weigh on a Bellinger type 2 pan balance of 2 to 5 kg capacity and an accuracy of 1 in 2 000. Dry the sample in an oven at  $105 \pm 5^\circ\text{C}$  to constant mass. It is recommended that the first mass to be taken after 3 h drying and subsequently after intervals of 1 h drying till constant mass is obtained. The weighing is done hot. Calculate moisture content as follows:

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\*Methods of sampling iron ores (*first revision*).

†Specification for water, for general laboratory use (*first revision*).



$$\text{Moisture, percent} = \frac{\text{Loss of mass in g}}{\text{mass in g of the sample taken}} \times 100$$

NOTE — For ores which are known to be of hygroscopic nature, samples shall be analysed on as received basis and the results reported on dry basis. For this purpose, moisture content of the sample shall be determined at the time of analysis and correction factor  $K$ , found out as follows, applied for each determination.

Dry a glass petridish in an air oven at  $105 \pm 5^\circ\text{C}$ . Cool in a desiccator and weigh. Transfer 5.00 g of the sample to the petridish and heat it in an air oven at  $105 \pm 2^\circ\text{C}$  to a constant mass. Cool in a desiccator and weigh. Calculate correction factor  $K$  as follows:

$$K = \frac{100}{100 - A}$$

where

$$A = \frac{\text{Loss in mass in g}}{5} \times 100$$

## 6. PREPARATION OF SAMPLE FOR CHEMICAL ANALYSIS

**6.1** The necessary portion of the sample required for chemical analysis is dried in an aluminium or glass petridish at  $105^\circ\text{C}$  for 1.5 to 2 h. The dish is cooled in a desiccator and from this dried sample the necessary portions required for different determinations are weighed.

## 7. DETERMINATION OF SILICA AND PREPARATION OF MAIN SOLUTION (FOR $\text{Si} \geq 0.5$ PERCENT)

**7.1 Outline of the Method** — The sample is dissolved in hydrochloric acid, and baked for dehydration of silica which is filtered and determined by volatilization with hydrofluoric acid. The insoluble residue is fused with sodium carbonate, extracted with hydrochloric acid and added to the main filtrate obtained on the first filtration. The solution is made up to a definite volume and reserved for further analysis (main solution).

### 7.2 Reagents

**7.2.1 Concentrated Hydrochloric Acid** — rd = 1.16 (conforming to IS : 265-1976\*).

**7.2.2 Concentrated Nitric Acid** — rd = 1.42 (conforming to IS : 264-1976†).

**7.2.3 Dilute Hydrochloric Acid** — 1 : 1 and 1 : 20 (v/v).

**7.2.4 Dilute Sulphuric Acid** — 1 : 1 and 1 : 20 (v/v).

**7.2.5 Hydrofluoric Acid** — 40 percent.

\*Specification for hydrochloric acid (second revision).

†Specification for nitric acid (second revision).

**7.2.6 Sodium Carbonate** — solid.

**7.2.7 Potassium Bisulphate** — solid.

### 7.3 Procedure

#### 7.3.1 For Ores Containing Titanium Dioxide $\leq 2$ percent

**7.3.1.1** Weigh accurately 2·500 g of the sample and transfer it to a 400-ml beaker. Add 35-40 ml of concentrated hydrochloric acid and 5 to 10 drops of concentrated nitric acid. Cover with watch-glass and digest on a hot-plate. Avoid boiling and stir the contents gently and occasionally. Continue digestion until all the ore is attacked and no black particles are left. Remove the watch-glass and wash with a little water.

**7.3.1.2** Evaporate the contents to dryness on low heat and bake the residue at about 120°C ( low in hot plate ) for 1 h. Cool somewhat and take up the residue in 10 ml of concentrated hydrochloric acid and dilute to about 20 ml. Warm the solution for dissolution ( at 70-80°C ) of the dried mass under cover. Filter the solution through Whatman filter paper No. 40 or equivalent and transfer all the insoluble matter from the beaker to filter paper using a policeman or rubber-tipped glass rod. Receive the filtrate in a 500-ml volumetric flask. Wash the residue with hot dilute hydrochloric acid ( 1 : 20 ) till the filter paper is free of ferric chloride. Finally wash the residue with hot water four or five times. Preserve the filtrate ( A ).

**7.3.1.3** Ignite the residue in a platinum crucible in a muffle furnace raising the temperature of the furnace slowly at the beginning and then maintaining the furnace at 1 050°C for about 30 min. Cool the residue in the platinum crucible in a desiccator and weigh. Moisten the residue with a little water and a few drops of dilute sulphuric acid ( 1 : 1 ) and 8 to 10 ml of hydrofluoric acid. Evaporate to dryness and repeat the procedure adding about 5 ml of hydrofluoric acid and weigh the platinum crucible. Calculate the percentage of silica ( 7·5 ).

**7.3.1.4** After determination of silica, fuse the residue left in the crucible with about 1 g of sodium carbonate. Extract the fused mass with dilute hydrochloric acid ( 1 : 1 ) and preserve the filtrate ( B ).

#### 7.3.2 For Ores Containing Titanium Dioxide More Than 2 Percent

**7.3.2.1** Weigh accurately 2·500 g of the sample and dissolve in concentrated hydrochloric acid as given in **7.3.1.1**. Add to the solution, 40 ml of dilute sulphuric acid ( 1 : 1 ). Evaporate the contents on a hot-plate or sand-bath until copious fumes of sulphuric acid are evolved. Cool thoroughly and dissolve the residue in 100 ml of water by boiling. Filter through a No. 40 filter paper and transfer the residue adhering to the beaker by means of a rubber-tipped glass rod. Wash the residue 7 or 8 times with hot dilute sulphuric acid ( 1 : 20 ). Preserve the filtrate ( A ).

**7.3.2.2** Dry and ignite the residue in a platinum crucible until all the carbon is burnt off. Cool and fuse the residue with 2 to 3 g of potassium bisulphate for about 5 min. Cool and extract the fused mass in a 400-ml beaker containing about 75 ml of water and 25 ml of dilute sulphuric acid ( 1 : 1 ). Wash the crucible and lid into the beaker. Evaporate the solution carefully and heat until copious fumes of sulphuric acid are evolved. Cool thoroughly and dissolve the residue in about 100 ml of water by boiling until all the soluble portion is dissolved. Filter the solution through a No. 40 filter paper and transfer the residue adhering to the beaker by means of a rubber-tipped glass rod. Wash the residue 7 or 8 times with hot dilute sulphuric acid ( 1 : 20 ). Preserve the filtrate ( *B* ). Treat the residue as directed in **7.3.1.3**.

**7.3.2.3** After the silica has been volatilized by treatment with hydrofluoric acid, fuse the residue in the platinum crucible with about 1 g of potassium bisulphate for about 5 min. Extract the fused mass in 60 to 70 ml of hot dilute sulphuric acid ( 1 : 20 ). Wash the crucible and lid into the same beaker. Preserve the solution ( *C* ).

**7.4 Preparation of Main Solution** — Combine the filtrate ( *A* ) obtained in **7.3.1.2** or **7.3.2.1**, the filtrate ( *B* ) obtained in **7.3.2.2** or **7.3.1.4** and the solution ( *C* ) obtained in **7.3.2.3** in the case of titaniferrous ores. Concentrate, if necessary, and transfer to a 500-ml volumetric flask, cool and make up to the mark and mix well. This is the solution of 2·500 g of the sample free from silica, which is hereafter referred to as the main solution. From this solution, the determinations of iron, alumina, titanium, manganese, calcium and magnesium are carried out.

## 7.5 Calculation

$$\text{Silica, percent} = \frac{A - B}{C} \times 100$$

where

- A* = mass of the crucible+residue before hydrofluorization,
- B* = mass of the crucible+residue after hydrofluorization, and
- C* = mass of the sample taken.

## 8. DETERMINATION OF IRON

**8.1 Outline of the Method** — Iron is reduced to ferrous state through silver reductor or by stannous chloride and the reduced iron is titrated with standard dichromate solution.

### 8.2 Reagents

**8.2.1 Silver Nitrate** — Solid.

**8.2.2 Concentrated Nitric Acid** — See **7.2.2**.

**8.2.3 Ammonium Chloride** — Solid.

**8.2.4 Ammonium Hydroxide** — rd=0·89.

**8.2.5 Methyl Red Indicator** — 0·5 percent.

**8.2.6 Ammonium Chloride Wash Liquid ( 5 percent )** — Ammonium chloride solution neutralized with ammonium hydroxide using methyl red indicator.

**8.2.7 Dilute Hydrochloric Acid** — 1 : 1, 1 : 4 ( v/v ), 1 *N* and 6 *N*.

**8.2.8 Dilute Phosphoric Acid** — 1 : 1 ( v/v ).

**8.2.9 Diphenylamine Sulphonate Indicator Solution** — Dissolve 0·5 g of sodium diphenylaminic sulphate in 100 ml water.

**8.2.10 Standard Potassium Dichromate Solution ( 0·1 *N* )** — Dissolve exactly 4·903 g of potassium dichromate ( dried at 100 to 110°C ) in water in a 1 000-ml volumetric flask and make up the volume up to the mark. Mix thoroughly and use as a standard solution.

**8.2.11 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 dissolution is complete. Cool and add a few pieces of granulated tin and preserve the solution in an air-tight amber-coloured bottle to prevent oxidation.

**8.2.12 Mercuric Chloride Solution** — Prepare a saturated solution of mercuric chloride in water.

**8.2.13 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.

### **8.3 Procedure ( by Silver Reductor Method )**

**8.3.1** Pipette out an aliquot equivalent to 0·25 g ( 50 ml ) from the main solution in a 400-ml beaker. Add about 1 ml of concentrated nitric acid and boil the solution for about 5 min. Cool somewhat, dilute to 150 ml volume and add about 3 g of ammonium chloride. Boil again and precipitate  $R_2O_3$  by dropwise addition of ammonium hydroxide adding methyl red indicator towards the end. Boil for a while for better coagulation and allow the precipitate to settle.

**8.3.2** Filter hot through Whatman filter paper No. 41 or equivalent and wash the residue 5 or 6 times with hot ammonium chloride wash liquid. Transfer back the precipitate to the original beaker, wash the filter paper with hot dilute hydrochloric acid ( 1 : 4 ) and collect the washings into the beaker.

**8.3.3** Dissolve the residue in the beaker with 10 ml of hot dilute hydrochloric acid ( 1 : 1 ) and 30-40 ml of water. Adjust the acid strength of the solution to about 1 *N* dilute hydrochloric acid by adding water. Cool the solution into room temperature and pass through silver reductor at the rate of 25-30 ml per minute as given in Appendix A. Collect the solution passing through the reductor in a 400-ml beaker. Wash the beaker and the reductor thoroughly with hydrochloric acid wash liquid ( 1 *N* ). ( Six washings of about 20 ml volume in each washing are sufficient. ) After collecting the washings, add 5 ml of dilute phosphoric acid and 5 or 6 drops of diphenylamine sulphonate indicator solution. Stir to mix properly and titrate with standard potassium dichromate solution till the violet blue colour persists.

#### 8.4 Calculation

$$\text{Iron, percent} = \frac{A \times B \times 0.05585 \times 100}{C}$$

where

*A* = volume in ml of standard dichromate solution consumed;

*B* = strength of the standard dichromate solution; and

*C* = mass in g of the sample taken, representing the aliquot.

#### 8.5 Procedure ( by Stannous Chloride Method ) ( Alternative Method )

**8.5.1** Pipette out 50 ml of the main solution and precipitate  $R_2O_3$  ( see **8.3.1** and **8.3.2** ). Dissolve the  $R_2O_3$  in 20 to 25 ml of dilute hydrochloric acid ( 6 *N* ) and transfer the solution to a 500-ml conical flask. Wash the beaker with a little water and transfer the washings to the conical flask. Heat to boiling for a while and add stannous chloride solution dropwise to the boiling solution with constant swirling until the yellow colour solution becomes just colourless. Add 2 or 3 drops excess of stannous chloride solution and wash the sides with a little water. Cool the flask rapidly under running tap water or otherwise until the contents have cooled down to room temperature.

**8.5.2** Add about 10 ml of mercuric chloride solution and shake the flask. At this stage a silky white precipitate appears. Wait for about 2 min, dilute to 150 to 170 ml volume and add 15 ml of sulphuric acid — phosphoric acid mixture. Add 3 or 4 drops of diphenylamine sulphonate indicator solution and titrate with standard potassium dichromate solution continuously until a stable violet blue colour persists.

**8.6 Calculation** — see 8.4.

**9. DETERMINATION OF PHOSPHORUS BY THE MOLYBDATE (ALKALIMETRIC) METHOD (FOR P  $\geq$  0.05 percent)**

**9.1 Outline of the Method** — From the sample solution,  $R_2O_3$  is precipitated by ammonium hydroxide.  $R_2O_3$  is dissolved in nitric acid. The phosphorus is precipitated as ammonium phosphomolybdate. The precipitate is filtered, washed and dissolved in excess of standard alkali solution. The unreacted alkali is back titrated against standard hydrochloric acid.

**9.2 Reagents**

**9.2.1 Dilute Ammonium Hydroxide Solution** — ( 1 : 1 ) ( v/v ).

**9.2.2 Dilute Nitric Acid** — 1 : 1 and 1 : 100 ( v/v ).

**9.2.3 Concentrated Nitric Acid** — see 7.2.2.

**9.2.4 Ammonium Nitrate** — solid.

**9.2.5 Nitro-Ammonium-Molybdate Solution**

- a) *Solution A* — Dissolve 100 g of molybdic acid or 118 g of ammonium molybdate in a mixture of 145 ml of concentrated ammonium hydroxide and 270 ml of water. Boil for a while and cool thoroughly.
- b) *Solution B* — Add 490 ml of concentrated nitric acid to 1 150 ml of water and cool. Add slowly solution *A* to solution *B* with constant stirring avoiding any appreciable rise in temperature. Add a pinch of ammonium phosphate and keep the solution at least for 24 h. Filter through No. 1 filter paper before use.

**9.2.6 Potassium Nitrate Wash Solution** — 1 percent ( m/v ).

**9.2.7 Sodium Hydroxide Solution** — 0.1 N.

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

**9.2.9 Standard Hydrochloric Acid ( 0.1 N )** — Dilute 11 ml of concentrated hydrochloric acid to 1 litre and standardize against standard sodium carbonate solution.

**9.3 Procedure**

**9.3.1** Pipette out 200 ml of the main solution in a 500-ml beaker, boil the solution and add with stirring dilute ammonium hydroxide solution until  $R_2O_3$  is precipitated. Add slight excess of ammonium hydroxide solution and boil for 2 to 3 min. Filter through number 40 filter paper, wash with hot water 5 or 6 times. Discard the filtrate.

**9.3.2** Dissolve the precipitate in dilute nitric acid ( 1 : 1 ) in the beaker and transfer the solution to a 500-ml glass stoppered conical flask. Wash the beaker and transfer the wash solution to the conical flask. Reduce the volume to about 75 ml. Add, if necessary, 5 ml of concentrated nitric acid and about 5 g of ammonium nitrate. Add with constant shaking 50 ml of nitro-ammonium-molybdate solution. Continue shaking till the precipitation is complete. Keep at 50 to 60°C for 1 h.

**9.3.3** Filter the precipitate through filter pad made out of Whatman filter paper No. 1 or 41. Wash the conical flask and the residue twice with dilute nitric acid solution ( 1 : 100 ) and then with potassium nitrate wash solution till 10 ml of the filtrate collected in a test tube does not consume more than one drop of sodium hydroxide solution, using a drop of phenolphthalein indicator solution. Transfer the precipitate with filter paper to the conical flask in which phosphorus was precipitated. Pulp the filter paper after adding 10 ml of sodium hydroxide solution. Wash the conical flask with a little water. Add 3 or 4 drops of phenolphthalein indicator solution. If there is no pink colour add more sodium hydroxide solution till the colour persists. Titrate with standard hydrochloric acid until the pink colour is just discharged.

**9.3.4** Carry out a blank on the same quantity of sodium hydroxide solution which was added in the test and titrate with standard hydrochloric acid, using 3 or 4 drops of phenolphthalein indicator solution.

#### 9.4 Calculation

$$\text{Phosphorus, percent} = \frac{(A - B) \times C \times 0.00135 \times 100}{D}$$

where

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

*B* = Volume in ml of standard hydrochloric acid required to titrate excess sodium hydroxide solution,

*C* = Normality of standard hydrochloric acid, and

*D* = Massing of the sample taken, representing the aliquot.

### 10. DETERMINATION OF ALUMINIUM BY COMPLEXOMETRIC METHOD

**10.1 Outline of the Method** — From the solution of the ore, iron is removed by MIBK extraction. After destroying the residual organic matter in the aqueous phase by nitric acid and  $\text{H}_2\text{O}_2$  the mixed oxides (  $\text{R}_2\text{O}_3$  ) are precipitated with ammonia.  $\text{R}_2\text{O}_3$  is filtered, dissolved in acid and poured into hot caustic soda solution. After filtration, aluminium in the filtrate is complexed with excess EDTA. The excess EDTA is titrated with zinc

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solution, using xylenol orange indicator. Aluminium complexed with EDTA is released by addition of ammonium fluoride and titrated with zinc solution.

### 10.2 Reagents

**10.2.1 Dilute Hydrochloric Acid** — 1 : 1, 5 : 3 and 3 : 1 ( v/v ).

**10.2.2 MIBK** ( *Methyl Isobutyl Ketone* )

**10.2.3 Concentrated Nitric Acid** — see 7.2.2.

**10.2.4 Hydrogen Peroxide, 30 percent** — 100 Vol.

**10.2.5 Ammonium Chloride** — solid.

**10.2.6 Methyl Red Indicator Ethanol Solution** — 0.5 percent.

**10.2.7 Ammonium Hydroxide** — 1 : 1 and 10 percent.

**10.2.8 Ammonium Nitrate Solution** — Slightly ammonical.

**10.2.9 Sodium Hydroxide Solution** — 10 percent ( v/v ).

**10.2.10 Sodium Carbonate** — anhydrous.

**10.2.11 EDTA Solution ( 0.01 M )** — Dissolve 3.72 g of the disodium salt of EDTA [  $(\text{OOC} \cdot \text{CH}_2)_2\text{NH} \cdot \text{CH}_2\text{CH}_2 \cdot \text{NH} ( \text{CH}_2\text{COO} )_2 \text{Na}_2 \cdot 2\text{H}_2\text{O}$  ] in water and transfer to a 1 000-ml volumetric flask, dilute to the mark and mix.

**10.2.12 Ammonium Acetate** — solid.

**10.2.13 Xylenol Orange Indicator** — Dissolve 0.25 g of xylenol orange in 100 ml of water containing two drops of dilute hydrochloric acid ( 1 : 1 ). The solution shall be prepared fresh every week.

**10.2.14 Standard Zinc Solution ( 0.01 M )** — Dissolve 0.653 8 g of electrolytic zinc granules ( 99.95 percent purity ) in dilute hydrochloric acid ( 1 : 1 ) and transfer the solution to a 1 000-ml volumetric flask, dilute with water to the mark and mix.

**10.2.15 Ammonium Fluoride Solution** — Dissolve 10 g of ammonium fluoride in 100 ml of water in a polythene beaker. Prepare fresh every day.

### 10.3 Procedure

**10.3.1** Pipette out an aliquot equivalent ( 0.50 g ) of the sample ( 100 ml aliquot ) from the main solution in a 250-ml beaker. Evaporate down to about 25 ml. Add 25 ml of dilute hydrochloric acid ( 3 : 1 ). Dissolve



the salts by gentle heating. Transfer to a 250-ml separatory funnel, add an equal volume of methyl isobutyl ketone. Extract for 1 min and let settle. Drain the aqueous phase into a 250-ml beaker and remove it. Wash the MIBK phase with 10 to 15 ml of dilute hydrochloric acid ( 5 : 3 ). Extract for 30 s. Let settle. Drain this aqueous phase into the beaker containing the reserved aqueous phase. Evaporate the aqueous phase down to about 10 ml. Add 5 ml of concentrated nitric acid and 2 ml of hydrogen peroxide. Heat to decompose the organic matter. Repeat the addition of concentrated nitric acid until the brown fumes cease to evolve. Heat to dryness and cool.

**10.3.2** Add 20 ml of dilute hydrochloric acid ( 1 : 1 ) and about 20 ml of hot water to ensure complete dissolution. Add 2 to 3 g of ammonium chloride and bring the contents to boiling. Add 2 or 3 drops of methyl red indicator and neutralize with dropwise addition of ammonium hydroxide ( 1 : 1 ) until the pink colour just turns yellow. Add 1 or 2 drops of ammonium hydroxide. Simmer the contents for 2 min. Add some filter paper pulp and filter hot through Whatman No. 41 or equivalent filter paper. Wash the filter paper 8 to 10 times with slightly ammonical ammonium nitrate solution.

**10.3.3** Dissolve the residue in dilute hydrochloric acid ( 1 : 1 ) and make the volume to about 50 ml. Slowly and while swirling constantly pour into a 200-ml volumetric flask containing 100 ml warmed sodium hydroxide solution to which has been added about 1 g of sodium carbonate. Warm on a water bath for about 1 h and cool to room temperature, dilute with water to the mark, mix well and let stand for a few minutes. Dry filter through filter paper Whatman No. 42, catching the filtrate in a beaker ( rinse the beaker first with a few ml of the solution and reject 5-10 ml of the filtrate ). Transfer 100 ml aliquot from the filtrate to a 250-ml conical flask. Add an excess of EDTA ( 25 ml is sufficient ). Add 3 or 4 drops of methyl red indicator solution and neutralize with dropwise addition of dilute hydrochloric acid ( 1 : 1 ) to just pink end point. Add 2 to 3 g of ammonium acetate. The pink colour changes to yellow. Boil the solution for about 5 min. Cool to room temperature and add 6 drops of xylenol orange indicator and titrate with standard zinc solution. The colour changes to pink at the end point.

**10.3.4** Add about 15 ml of ammonium fluoride solution and heat the contents to boiling. Boil gently for about 5 min. Cool thoroughly. Add about 1 g of ammonium acetate and titrate the liberated EDTA with zinc solution before adding about 3 drops of xylenol orange indicator solution. Record the titre in ml.

#### 10.4 Calculation

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

where

$A$  = Volume in ml of standard zinc solution required for the titration of liberated EDTA,

$B$  = equivalent aluminium in mg per ml of standard zinc solution, and

$C$  = mass in mg of the sample represented by the aliquot of solution taken.

## 11. DETERMINATION OF SULPHUR

**11.1 Outline of the Method** — The sample is decomposed with potassium chlorate and hydrochloric-nitric acid mixture, and the solution is evaporated to dryness after adding hydrochloric acid. The soluble salt is dissolved in hydrochloric acid, and the insoluble residue is filtered off. Bulk of the iron in the filtrate is extracted with methyl isobutyl ketone, and organic matter is decomposed with the acid mixture ( the main solution ). Silica is volatilized from the insoluble residue with hydrofluoric acid, the non-volatile matter is fused with sodium carbonate and the melt is leached with warm water and filtered. The carbonate in the filtrate is acidified and combined with the main solution.

**11.1.1** Zinc is added to reduce the remaining trivalent iron to bivalent iron, and acidity is adjusted.

**11.1.2** Barium chloride solution is added and the barium sulphate precipitate is filtered off. The precipitate is ignited until constant mass is obtained, and the amount of sulphur is calculated from the mass.

### 11.2 Reagent

NOTE — Distilled or deionized water shall be used in the preparation of reagents and throughout the analysis. All reagents shall be of recognized analytical reagent quality.

**11.2.1 Potassium Chlorate** — Powder.

**11.2.2 Acid Mixture** — Hydrochloric acid ( 4 ) + nitric acid ( 1 ).

**11.2.3 Concentrated Hydrochloric Acid** — see 7.2.1.

**11.2.4 Dilute Hydrochloric Acid** — 10 : 5, 1 : 1, 2 : 100.

**11.2.5 Ammonium Thiocyanate Solution** (  $NH_4CNS$  ) — 100 g/l ( m/v ).

**11.2.6 Methyl Isobutyl Ketone** — Saturate with dilute hydrochloric acid as follows:

Place methyl isobutyl ketone and dilute hydrochloric acid ( 2 : 1 ) into a 300-ml separating funnel, and shake thoroughly for about 1 min. Allow to stand until the layers separate, discard the lower aqueous layer and use the remaining solution.

**11.2.7 Sodium Nitrate Solution** — Saturated solution in water.

**11.2.8 Concentrated Nitric Acid** — see 7.2.2.

**11.2.9 Hydrofluoric Acid** — 40 percent and rd — 1·13 g/ml

**11.2.10 Sodium Carbonate** — Solid.

**11.2.11 Sodium Carbonate Solution** ( 20 g  $Na_2CO_3/1$  ) — Store in a plastic bottle.

**11.2.12 Methyl Orange Solution** — Dissolve 0·10 g of methyl orange in 100 ml of water.

**11.2.13 Zinc** — Use zinc with the lowest content of sulphur and of 1-3 mm size.

**11.2.14 Barium Chloride Solution** ( 100 g/l ) — Dissolve 100 g of crystalline barium chloride (  $BaCl_2$  ) in 1 litre of water, cover and allow the solution to cool overnight to room temperature. Store the solution in a plastic bottle and filter through a close texture filter paper the required volume before each use.

**11.2.15 Hydrochloric Acid Wash Solution Containing Barium Chloride** — Filter 10 ml of the barium chloride solution ( 11·2·14 ) through a close texture filter paper, and dilute with 1 litre of dilute hydrochloric acid ( 2 : 100 ).

**11.2.16 Silver Nitrate Solution** ( 10 g  $AgNO_3/1$  ) — ( m/v ).

### 11.3 Procedure

**11.3.1 Decomposition of Test Portion** — Weigh 5·0 g of the sample. Place the test sample portion in a 500-ml beaker, add 1 g of potassium chlorate and 3-4 ml of water, and shake thoroughly. Add 70 ml of acid mixture and heat gently to decompose the ore. Evaporate the solution on a hot-plate heated to 110 to 120C to dryness and cool.

**11.3.2** Add 30 ml of dilute hydrochloric acid ( 1 : 1 ) and dissolve soluble salts by warming. Add 25 ml of water and boil for about 5 min. Filter the solution through a close texture filter paper and wash the paper with warm dilute hydrochloric acid ( 2 : 100 ) until iron ion is no longer detected in the washings by test with ammonium thiocyanate solution.

#### 11.3.3 Extraction of the Bulk of the Iron

**11.3.3.1** Collect the filtrate and washings in a 500-ml beaker, and evaporate by heating to about 10 ml. Transfer the solution to a 200-ml

separating funnel with 50 ml of dilute hydrochloric acid ( 10 : 5 ) in small portions. Add 50 ml of methyl isobutyl ketone, shake thoroughly for about 1 min, and allow to stand until the layers separate. Draw off the aqueous layer into another 200-ml separating funnel. Wash the remaining organic layer by adding 10 ml of dilute hydrochloric acid ( 10 : 5 ) and allow to stand until the layers separate. Draw off and collect the aqueous layer into the latter separating funnel. Wash again the organic phase with another 10 ml of dilute hydrochloric acid ( 10 : 5 ) and collect the aqueous solution.

**11.3.3.2** Add once again to the separating funnel 50 ml of methyl isobutyl ketone and shake thoroughly to complete the extraction. Draw off the lower aqueous solution into a 500-ml beaker, wash the organic layer by adding 10 ml of dilute hydrochloric acid ( 10 : 5 ) and transfer the washings to the beaker. Wash again the organic phase with another 10 ml of dilute hydrochloric acid ( 10 : 5 ) and collect the aqueous solution. Evaporate the aqueous solution by heating near to dryness.

**11.3.3.3** Add 10 ml of acid mixture and evaporate to dryness, decomposing the organic matter. Add 10 ml of concentrated hydrochloric acid and evaporate to dryness on a hot-plate heated to about 150°C. Add further 10 ml of concentrated hydrochloric acid, evaporate to dryness, allow to heat at 110°C and cool. Add 5 ml of concentrated hydrochloric acid and 30 ml of water to dissolve the residue on evaporation, filter and preserve as the main solution.

**11.4 Treatment of Residue** — The procedure to be adopted if any insoluble sulphate is expected.

**11.4.1** Place the residue with the filter paper in a platinum crucible, moisten the filter paper with 1 or 2 drops of sodium nitrate solution, heat the crucible carefully and ignite at 800 to 850°C. Allow the crucible to cool, moisten the ignition residue with a few drops of concentrated nitric acid, add about 5 ml of hydrofluoric acid and heat gently to remove silicon dioxide and nitric acid.

**11.4.2** Allow the crucible to cool, add 3 g of sodium carbonate and fuse the residue at  $1000 \pm 50^\circ\text{C}$  for about 15 min. Allow the crucible to cool, place the crucible containing the melt in a 300-ml beaker, add about 100 ml of warm water and heat to dissolve the melt. Take out the crucible and rinse with water.

**11.4.3** Collect the residue on a close texture filter paper, wash the filter paper several times with sodium carbonate solution, and discard the residue. Combine the filtrate and washings, add dilute hydrochloric acid ( 1 : 1 ) in small portions, while stirring to neutralize the solution using 0.5 ml methyl orange solution as the indicator. Add 5 ml of dilute hydrochloric acid ( 1 : 1 ) in excess and combine with the main solution from **11.3.3.3**.

## 11.5 Precipitation of Barium Sulphate

**11.5.1** Cover the beaker containing the combined main solution with a watch-glass, and heat to evaporate the solution to 25 ml. Allow the solution to cool, add 3 ml of dilute hydrochloric acid ( 1 : 1 ) and 50 ml of water and heat the solution to dissolve soluble salts. Add 1.0 g of zinc in the portion, while heating on a water bath at 60-70°C, to reduce the remaining trivalent iron to bivalent iron. Filter the solution through a close texture filter paper immediately after complete dissolution of the zinc, and wash with dilute hydrochloric acid ( 2 : 100 ) to a total volume of 120 ml. Collect the filtrate and washings in a 300-ml tall form beaker. Discard the residue.

**11.5.2** Boil the solution. Add slowly 5 ml of barium chloride solution from a burette. Continue boiling for 5 min and then keep in a steam bath for 2 h or preferably overnight.

**11.5.3** Collect the precipitate of barium sulphate on a close texture filter paper and wash the beaker once with hydrochloric acid wash solution containing barium chloride. Scrap out the precipitate still adhering to the walls of the beaker using a rubber-tipped glass rod, transfer to the filter paper with the aid of hydrochloric acid wash solution containing barium chloride and wash out the beaker several times with the same solution. Wash the precipitate on the filter paper with warm water until chloride ion is no longer detected in the washings by test with silver nitrate solution.

## 11.6 Weighing

**11.6.1** Place the precipitates from **11.5.3** with the filter paper in a tared platinum crucible. Dry at low temperature, ignite to ash carefully at about 500°C. Add 1 or 2 drops of sulphuric acid ( 1 : 1 ) and ignite at about 800°C for about 20 min until constant mass is obtained. Allow the crucible to cool to room temperature in a desiccator and weigh as barium sulphate.

## 11.7 Expression of Results

**11.7.1** *Calculation of Sulphur Content* — The sulphur content, as a percentage by mass, is calculated from the following:

$$\text{Sulphur, percent ( m/m )} = \frac{(m_1 - m_2) \times 0.1374 \times 100}{m_3}$$

where

$m_1$  = mass in g of the platinum crucible with barium sulphate,

$m_2$  = mass in g of the platinum crucible, and

$m_3$  = mass in g of the test portion.

**11.7.2** *Blank Value* — Carry out a blank test and subtract the blank value from the analytical value.

## APPENDIX A

( Clause 8.3.3 )

## PREPARATION OF SILVER REDUCTOR

Take 30 g of silver nitrate in a 600-ml beaker, dissolve in 400 ml of water. Acidify with about 10 ml of concentrated nitric acid (  $\rho_d=1.42$  ) Immerse in the solution crosswise two rods of Zn each about 15 cm long, 5-6 mm in diameter. Leave the whole thing overnight. Remove the rods and decant off the solution. Wash the silver thoroughly by decantation with dilute sulphuric acid. Then transfer the whole thing to the silver reductor column. The reductor column has the main portion made of pyrex glass tube of 12 cm length and 2 cm diameter. There is a glass stopper at the bottom and the end is drawn to narrow tip. Above the stopper is placed a perforated ceramic disc and some glass wool. ( see Fig. 1 ). The upper part of the column is made into a wider beaker like cup of about 6 cm diameter. Transfer the silver to the column ( about 10 cm ), wash with dilute sulphuric acid taking care that no bubbles or air gaps are formed in the column. Drain out the solution and finally wash several times with dilute hydrochloric acid ( 1 *N* ) before it is used for reduction.

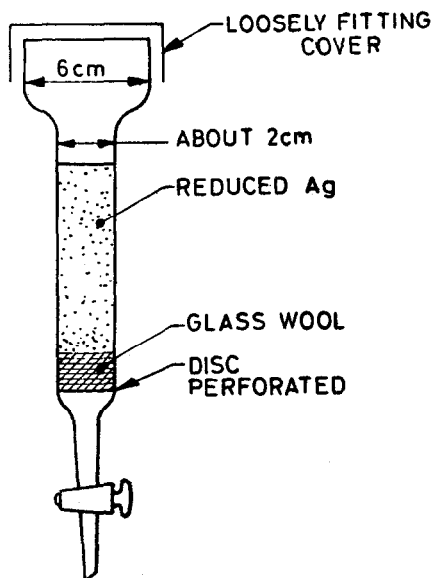


FIG. 1 SKETCH OF SILVER REDUCTOR

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