

**SRI LANKA STANDARD 676:1984**  
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**METHODS OF TEST FOR  
HEAVY MINERALS**

**SRI LANKA STANDARDS INSTITUTION**



# METHODS OF TEST FOR HEAVY MINERALS

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SRI LANKA STANDARD  
METHODS OF TEST FOR HEAVY MINERALS

**FOREWORD**

This Sri Lanka Standard was authorized for adoption and publication by the Council of the Sri Lanka Standards Institution on 1984-12-20 after the draft, finalized by the Drafting Committee on Mineral Sands, had been approved by the Chemicals Divisional Committee.

The heavy mineral beach sand deposits found in Sri Lanka contain large concentrations of ilmenite, rutile and monazite. Importance of mineral sands in the export market is well recognized. In order to determine correctly the chemical composition of heavy minerals, need was felt to prescribe standard methods of test so that these may primarily serve as referee methods and may also be used by laboratories for routine analysis.

All standard values given in this standard are in SI units.

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 CS 102.

In the preparation of this standard, valuable assistance derived from the relevant publications of the Indian Standards Institution is gratefully acknowledged.

**1 SCOPE**

This standard prescribes the methods of test for heavy minerals. It covers the determination of the following;

- a) Titania
- b) Silica
- c) Iron (III) oxide
- d) Iron (II) oxide
- e) Total iron

- f) Zirconia
- g) Alumina
- h) Phosphorous pentoxide
- j) Chromium oxide
- k) Vanadium oxide
- m) Moisture content
- n) Grain size distribution.

## 2 REFERENCES

- CS 102 Presentation of numerical values
- CS 124 Test sieves
- SLS 677 Sampling of heavy minerals

## 3 SAMPLING

3.1 Representative samples of the materials for carrying out tests shall be drawn as prescribed in SLS 677.

## 4 GENERAL

4.1 During the analysis, unless otherwise stated use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.2 In the methods prescribed in this standard only the relative number of Whatman filter papers have been prescribed. However, any other suitable brand of filter paper of corresponding porosity may be used.

## 5 DETERMINATION OF TITANIA

For the determination of titania, the routine method given in 5.1 shall be used. But in case of dispute, the reference method given in 5.2 shall be used.

### 5.1 Routine method : Reduction (Volumetric) method

#### 5.1.1 *Outline of the method*

5.1.1.1 After reduction with zinc amalgam, titanium is determined volumetrically by titrating with standard ammonium iron (III) sulfate solution using potassium thiocyanate as indicator.

#### 5.1.2 *Apparatus*

5.1.2.1 The apparatus for reduction (Figure 1) consists of a globular separating funnel A with a rubber stopper B carrying a tube G with a stop cock H. Flask C of 50-ml capacity is attached to the lower end



of the separating funnel by means of a pressure tubing D having a pinch cock E. Any other similar apparatus may also be used.

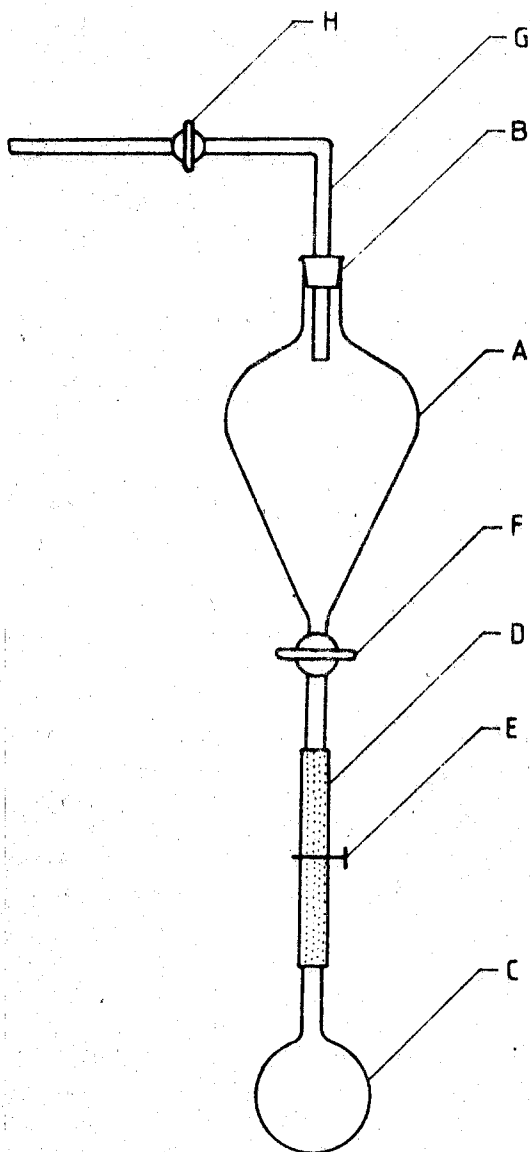


FIGURE 1 - Apparatus for determination of titania by the reduction (volumetric) method

### 5.1.3 Reagents

5.1.3.1 Sulfuric acid, dilute, one per cent (V/V) and 20 per cent (V/V), prepared by using concentrated sulfuric acid (relative density = 1.84).

5.1.3.2 Carbon dioxide, gas.

5.1.3.3 Potassium dichromate, standard volumetric solution,  $c(\text{K}_2\text{Cr}_2\text{O}_7) = 0.008 \text{ mol/l}$ .

Dry, potassium dichromate crystals at  $105^\circ \text{C}$  for one and a half hours. Cool in a desiccator and transfer 2.354 g to a one-litre volumetric flask carefully, through a funnel using water at room temperature. Wash the funnel thoroughly and dissolve the salt by shaking until the solution is complete. Make up to the mark and mix well.

5.1.3.4 Ammonium iron (III) sulfate, standard volumetric solution,  $c [\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}] =$  approximately 0.05 mol/l.

a) Dissolve 24 g of ammonium iron (III) sulfate in 350 ml of water containing 40 ml of concentrated sulfuric acid. Add a solution of potassium permanganate dropwise until the first appearance of a permanent trace of pink colour, filter through glass wool and dilute to one litre.

b) Reduce 25 ml of the above solution with zinc amalgam (5.1.3.6) following the procedure given in 5.1.4.1 but using the above solution in place of the sample (i.e. in place of filtrate preserved under 6.3.5). Add 10 ml of 85 per cent (V/V) ortho phosphoric acid (relative density = 1.69) and 8 drops of one per cent sodium diphenylamine sulfonate indicator solution and titrate against potassium dichromate solution (5.1.3.3) while swirling the flask until the colour changes to port wine red.

5.1.3.5 Potassium thiocyanate, 5 per cent solution (m/V).

5.1.3.6 Zinc amalgam, wash 15 g of granulated zinc with one per cent (V/V) sulfuric acid and heat with 300 g of pure mercury after adding 5 ml of 20 per cent (V/V) sulfuric acid, for one hour on a water bath in a fume cupboard. (Caution : Mercury vapours are highly poisonous). Wash the amalgam several times with 50 per cent (V/V) sulfuric acid after cooling and separate the liquid portion from the solids in a separating funnel. Preserve the liquid amalgam under 20 per cent (V/V) sulfuric acid.

#### 5.1.4 Procedure

5.1.4.1 Fill the flask C, the rubber tubing D and the stem upto the stop-cock F with freshly boiled and cooled water containing one per cent of sulfuric acid. Close pinch-cock E and the stop-cock F. Pipette 50-ml of the filtrate preserved under 6.3.5 into the separating funnel A containing 50 ml of zinc amalgam (5.1.3.6). Pass carbon dioxide gas (5.1.3.2) into the separating funnel for 5 minutes through G. Stopper the funnel, close stop-cock H and shake it for 5 minutes. Open stop-cock F and pinch cock E and transfer amalgam into the flask C by pressing and releasing the pressure tubing D. Close the pinch-cock E and stop-cock F immediately after the last drop of amalgam has passed into the flask C. Disconnect the flask C from the pressure tubing D. Remove the stopper B and wash it with boiled and acidified water into the separating funnel.

5.1.4.2 Add 5 drops of potassium thiocyanate solution (5.1.3.5) into the separating funnel and titrate rapidly with the ammonium iron (III) sulfate solution (5.1.3.4) until the appearance of a pale red tinge. Open stop-cock F and squeeze the rubber tubing to force the liquid into the separating funnel. Complete titration by adding ammonium iron (III) sulfate solution drop by drop until the pale red tinge is permanent.

5.1.4.3 Repeat the experiment with another 50-ml aliquot following the procedure given in 5.1.4.1 and 5.1.4.2 and adding nearly whole of the volume of ammonium iron (III) sulfate solution required for the previous titration at the beginning of the titration. Agitate by shaking with a gentle rotary motion and continue until the final end point is reached.

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

### 5.1.5 Calculation

$$\text{Titania, per cent by mass, (see Note)} = \frac{(V_1 - V_2) \times c \times 7.99}{m}$$

where,

$V_1$  = volume, in millilitres, of the ammonium iron (III) sulfate solution used for the test solution;

$V_2$  = volume, in millilitres, of the ammonium iron (III) sulfate solution used for the blank;

$c$  = concentration of the ammonium iron (III) sulfate solution in moles per litre; and

$m$  = mass, in grams, of the sample represented by the aliquot taken.

*NOTE - Any traces of iron which might have been co-precipitated in the process will also be reported as titania. The exact value of titania content could be calculated, if necessary, by determining the iron content of the solution in the separating funnel, as iron (III) oxide in accordance with 7 or any instrumental method.*

## 5.2 Reference method

### 5.2.1 Outline of the method

5.2.1.1 Titanium is precipitated quantitatively in acid solution in the presence of ammonium oxalate, as a flocculent, orange-red, titanium-tannin complex by the reagent tannin. This precipitate, after washing free of excess reagent and salts, is ignited to give a precipitate of titanium dioxide ( $\text{TiO}_2$ ) and weighed.

### 5.2.2 Reagents

5.2.2.1 Potassium bisulfate, solid.

5.2.2.2 Ammonium oxalate, saturated solution.

5.2.2.3 Ammonium hydroxide, 25 per cent (V/V) solution, prepared by using concentrated ammonium hydroxide (relative density = 0.88)

5.2.2.4 Hydrochloric acid, 50 per cent (V/V) solution, prepared by using concentrated hydrochloric acid (relative density = 1.18)

5.2.2.5 Hydrochloric acid, 9 per cent (V/V) solution, prepared by using concentrated hydrochloric acid (relative density = 1.18)

5.2.2.6 Hydrochloric acid, 30 per cent (V/V) solution, prepared by using concentrated hydrochloric acid (relative density = 1.18)

5.2.2.7 Tannin, 10 per cent (m/V) solution.

5.2.2.8 Ammonium chloride, 2 per cent (m/V) solution.

5.2.2.9 Ammonium hydroxide, 7 per cent (V/V) solution, prepared by using concentrated hydrochloric acid (relative density = 0.88)

5.2.2.10 Ammonium chloride, saturated solution.

### 5.2.3 Procedure

5.2.3.1 Weigh to the nearest milligram about 0.2 grams of the finely powdered sample. Fuse the sample with 3 g of potassium bisulfate (5.2.2.1) in a silica crucible, until crystals of neutral potassium sulfate separate at the surface of the fluid melt. Cool the crucible, add ammonium oxalate solution (5.2.2.2) and warm gently to detach the melt. Transfer the contents of the crucible to a 250-ml beaker with hot water and add more ammonium oxalate solution (75 ml in all). Filter the solution into a 600-ml beaker and wash the filter paper with hot water.

#### 5.2.3.2 Major titania fraction

To the above filtrate (5.2.3.1) whose volume is kept at about 150-ml (reduced to this volume by evaporation if necessary), add ammonium hydroxide (5.2.2.3) dropwise until a faint turbidity persists and then add 6 drops of hydrochloric acid (5.2.2.4) and dilute with an equal volume (about 150-ml) of saturated ammonium chloride (5.2.2.10) and boil. Precipitate the titanium by adding 30 ml of freshly prepared solution of tannin (5.2.2.7) with brisk agitation. Continue boiling for one minute and collect the voluminous red precipitate on a No. 40 Whatman filter paper containing some filter-fibre, under slight suction. Transfer the filtrate to a 800-ml beaker and boil down. Transfer the precipitate to the original beaker with a jet of hot ammonium chloride wash liquor (5.2.2.8) (150-ml) and add 1 to 2 drops of hydrochloric acid (5.2.2.4). Churn the suspension well with a glass rod until the clots are broken up. Filter the suspension through the same filter paper. Clean the beaker with the aid of filter pulp and complete the washing. Subject the filter and precipitate to strong suction and transfer into a tared platinum (or porcelain) crucible and heat gently until well charred. Complete the ignition on a Meker's burner with the crucible on a silica triangle. The resulting titania residue ( $\text{TiO}_2$ ) is preserved.

### 5.2.3.3 Minor titania fraction

a) Add the washings of the precipitate in 5.2.3.2 to the concentrated filtrate in 5.2.3.2. Treat this with 10 ml of tannin solution while boiling, followed by dropwise addition of ammonium hydroxide (5.2.2.9) with constant stirring. Each drop of ammonium hydroxide will produce a transient darkening of the liquid at the point of incidence due to the action of tannin on the iron (III) ions present and finally the liquid assumes a permanent slight mauve tint while a small orange precipitate flocculates. Leave this precipitate to settle and collect in the same manner as the precipitate mentioned in 5.2.3.2 and ignite separately. If the residue is not perfectly white after ignition, fuse with potassium bisulfate (5.2.2.1) and re-treat by the same process for the removal of the small amount of co-precipitated iron.

b) The filtrate obtained, is normally free of titanium, but should always be tested for complete precipitation by treating at boiling point with ammonium hydroxide (5.2.2.9) until incipient iron precipitation is indicated by a violet cloudiness and then just clearing this by continuous addition of hydrochloric acid (5.2.2.5). If no orange flocks are visible, then all titanium has been recovered, otherwise the small flocculated precipitate shall be collected and added to the bulk precipitate before igniting.

Transfer the ignited titania fractions to a 50-ml beaker and digest with 10 ml of hydrochloric acid (5.2.2.6) for 30 minutes on a water bath. Make the liquid ammoniacal and collect the oxide on a 90-mm ashless filter paper. Wash this with the ammonium chloride solution (5.2.2.8) and ignite in a tared crucible and weigh.

### 5.2.4 Calculation

$$\text{Titania per cent by mass} = \frac{m_2 - m_1}{m_0} \times 100$$

where,

$m_0$  = mass, in grams, of the sample taken;

$m_1$  = mass, in grams, of the crucible; and

$m_2$  = mass, in grams, of the crucible and the final precipitate ( $\text{TiO}_2$ ).

## 6 DETERMINATION OF SILICA

### 6.1 Outline of the method

6.1.1 The sample is fused with potassium bisulfate. The melt is extracted with dilute sulfuric acid, warmed and filtered. The residue after fusion with sodium carbonate, is treated with dilute sulfuric acid and evaporated to fumes and filtered. Silica is determined by the difference in mass after volatilization with hydrofluoric acid in the presence of sulfuric acid.

## 6.2 Reagents

6.2.1 *Potassium bisulfate*, solid.

6.2.2 *Sulfuric acid, dilute*, 3 per cent (V/V), 10 per cent (V/V) and 50 per cent (V/V) solutions, prepared by using concentrated sulfuric acid (relative density = 1.84).

6.2.3 *Sodium carbonate*, solid.

6.2.4 *Hydrofluoric acid*, 40 per cent (V/V) solution, prepared by using concentrated hydrofluoric acid (relative density = 1.15)

6.2.5 *Hydrogen peroxide*, 3 per cent (m/V) solution.

## 6.3 Procedure

6.3.1 Weigh to the nearest 0.0001 g about 0.5 g of the sample. Transfer it to a silica crucible containing 6 g to 7 g of potassium bisulfate (6.2.1) and heat at a low temperature till potassium bisulfate is melted. Increase the temperature to dull red heat. As soon as the sample is completely decomposed remove the crucible from the heat and allow it to cool to room temperature.

6.3.2 Transfer the crucible with its contents to a beaker and extract the contents with 100 ml of 10 per cent sulfuric acid (6.2.2). Warm the solution and filter through a No. 40 Whatman filter paper. Remove any adhering particles from the beaker by means of 3 per cent sulfuric acid (6.2.2). Wash the precipitate and the filter paper with hot water until free from sulfate (test with barium chloride), collecting the filtrate and the washings in a 250-ml volumetric flask.

6.3.3 Transfer the filter paper with the residue to a platinum crucible, ignite and fuse it with 2 g of sodium carbonate (6.2.3). Extract the melt with about 25 ml of 50 per cent sulfuric acid (6.2.2) in the basin, add 10 ml of hydrogen peroxide (6.2.5) and evaporate to dense white fumes. Continue the fuming for 30 minutes. Cool and dilute to 50 ml with hot water and filter through a No. 40 Whatman filter paper and wash as in 6.3.2. Transfer the filtrate to the same 250-ml volumetric flask (see 6.3.2).

6.3.4 Ignite the filter paper along with the residue obtained in 6.3.3 in a platinum crucible at 1050 °C until the difference between two successive weighings is not more than 0.0001 g. Cool in a desiccator and weigh. Moisten the residue with a few drops of 50 per cent sulfuric acid (6.2.2) and add 5 ml to 10 ml of hydrofluoric acid (6.2.4). Evaporate to dryness, ignite, cool and weigh.

6.3.5 Fuse this residue with one gram of potassium bisulfate (6.2.1) and extract with 20 ml to 30 ml of 50 per cent sulfuric acid. If the solution is clear, transfer it to the same 250-ml volumetric flask (see 6.3.2 and 6.3.3) and make up the volume to 250-ml. If there is any precipitate due to zirconium phosphate then filter and wash. Fuse the precipitate with sodium carbonate (6.2.3). Digest the melt in 50 ml of hot water and filter. Wash with hot water and discard the filtrate. Dissolve the precipitate in hot 50 per cent sulfuric acid and add to the filtrate in the 250-ml volumetric flask, and make up the volume to 250 ml. Preserve the solution for determination of titanium dioxide, iron (III) oxide and alumina.

#### 6.4 Calculation

$$6.4.1 \text{ Silica, per cent by mass} = \frac{m_1 - m_2}{m_0} \times 100$$

where,

$m_0$  = mass, in grams, of the sample taken;

$m_1$  = mass, in grams, of the crucible and its contents before evaporating with hydrofluoric acid; and

$m_2$  = mass, in grams, of the crucible and its contents after evaporation with hydrofluoric acid.

### 7 DETERMINATION OF IRON (III) OXIDE

#### 7.1 Outline of the method

7.1.1 The iron (III) state is reduced to iron (II) state with hydroxylammonium chloride. Then the iron (II) is complexed with *o*-phenanthroline monohydrate in tri-sodium citrate buffer to form an orange-red coloured complex. The intensity of the colour is measured at 508 nm.

#### 7.2 Reagents

7.2.1 *Hydroxylammonium chloride*, 10 per cent (m/V) solution.

7.2.2 *Tri-sodium citrate*, 10 per cent (m/V) solution.

7.2.3 *o*-phenanthroline monohydrate, 0.1 per cent (m/V) solution

7.2.3.1 Dissolve 0.5 g of *o*-phenanthroline monohydrate by boiling in water and dilute the solution to 500-ml after cooling.

7.2.4 *Iron standard solution*

7.2.4.1 Dissolve 0.140 0 g of electrolytic iron or iron wire in 10 ml of concentrated hydrochloric acid (relative density = 1.18) by boiling. Transfer the solution to a one-litre volumetric flask and make up to the mark. This is the standard stock solution.

7.2.4.2 Dilute 100 ml of this stock solution to 1 000 ml with water. This is the working solution. 1 ml of this working solution contains 0.02 mg of iron (III) oxide.

7.2.5 *Ammonium hydroxide*, 50 per cent (V/V) solution, prepared by using concentrated ammonium hydroxide (relative density = 0.88).

7.2.6 *Hydrochloric acid*, 50 per cent (V/V) solution, prepared by using concentrated hydrochloric acid (relative density = 1.18).

### 7.3 Procedure

7.3.1 Pipette out exactly 10 ml of the solution preserved in 6.3.5 into a 100-ml beaker. Neutralize by adding ammonium hydroxide solution (7.2.5) until turbidity appears. Then add hydrochloric acid (7.2.6) until the turbidity just disappears. Add 10 ml of the hydroxylammonium chloride (7.2.1) to the neutralized solution and allow the solution to stand for five minutes. Add 10 ml of tri-sodium titrate solution (7.2.2) and 10 ml of *o*-phenanthroline monohydrate solution (7.2.3). Then transfer the solution to a 100-ml volumetric flask quantitatively, make up the solution to the mark and allow it to stand for two hours. Measure the transmittance in a spectrophotometer at 508 nm against reagent blank (7.3.2).

7.3.2 Prepare reagent blank following the same procedure and using the same amount of all reagents but without the sample.

7.3.3 To a series of 100-ml volumetric flasks add 1.0 ml, 2.0 ml, 3.0 ml, 4.0 ml, 5.0 ml, 6.0 ml, 7.0 ml, 8.0 ml, 9.0 ml and 10.0 ml of the working solution (7.2.4) and develop the colour using the same procedure and using the same amounts of all reagents. Make the solution upto the mark and measure the transmittance at 508 nm against reagent blank.

Plot transmittance values against iron concentrations to obtain a calibration graph.

### 7.4 Calculation

7.4.1 Total iron expressed as iron (III) oxide, per cent

$$\text{by mass} = \frac{m_1}{10 m_2}$$

where,

$m_1$  = mass, in milligrams, of iron (III) oxide as obtained from the calibration graph corresponding to the value of transmittance measured; and

$m_2$  = mass, in grams, of the sample represented by the aliquot taken.



## 8 DETERMINATION OF IRON (II) OXIDE

### 8.1 Outline of the method

8.1.1 The sample is brought into solution under non-oxidizing conditions. Iron (II) is then determined as iron (II) oxide by titrating against standard potassium permanganate solution.

### 8.2 Reagents

8.2.1 *Hydrofluoric acid*. 40 per cent (V/V) solution, prepared by using concentrated hydrofluoric acid (relative density = 1.15).

8.2.2 *Sulfuric acid*, 50 per cent (V/V) solution, prepared by using concentrated sulfuric acid (relative density = 1.84).

8.2.3 *Boric acid*, saturated solution.

8.2.3.1 Prepare a saturated solution of boric acid using approximately 2.5 g per 100 g of water.

8.2.4 *Potassium permanganate*, standard volumetric solution,  $c(\text{KMnO}_4) = \text{approximately } 0.02 \text{ mol/l}$ .

8.2.4.1 Weigh 3.20 g to 3.25 g of potassium permanganate on a watch glass, transfer it to a 1 500-ml beaker, add one litre of water, cover the beaker with a watch glass and boil gently for 15 to 30 minutes. Allow the solution to cool to room temperature. Filter through glass-wool and store in a glass-stoppered amber coloured bottle. Standardize the solution against arsenious oxide in the following manner:

Weigh to the nearest 0.001 g about 0.25 g of dried arsenious oxide into a 400-ml beaker. Add 10 ml of 20 per cent (m/V) sodium hydroxide while stirring and allow to stand for about 8 to 10 minutes. After the solution is complete, add 100 ml of water and 10 ml of concentrated hydrochloric acid and one drop of 0.4 g/l potassium iodide solution and titrate until the pink colour persists.

### 8.3 Procedure

8.3.1 Weigh to the nearest 0.000 1 g about 0.5 g of the sample into a platinum crucible. Add 10 ml of hydrofluoric acid (8.2.1) and 10 ml of sulfuric acid (8.2.2), cover with the lid and boil over a low yellow flame for about 30 minutes until thick white copious fumes are evolved. Immerse the crucible with the lid in a 600-ml beaker containing 400-ml of saturated boric acid solution (8.2.3). Titrate the solution against potassium permanganate solution (8.2.4), until a faint pink colour persists.

## 8.4 Calculation

$$8.4.1 \text{ Iron (II) oxide, per cent by mass} = \frac{7.185 \times c \times V}{m}$$

where,

$c$  = concentration of potassium permanganate solution in moles per litre;

$V$  = volume, in millilitres, of potassium permanganate required for the titration in 8.3.2; and

$m$  = mass, in grams, of the sample taken.

## 9 DETERMINATION OF TOTAL IRON

### 9.1 Outline of the method

9.1.1 The iron (III) state is reduced to iron (II) state by passing the solution through a column containing sponge silver (silver reductor). Iron (II) is then titrated against standard potassium dichromate solution to an end-point of port-wine red colour using the sodium salt of *N*-methyl diphenyl amine *p*-sulfonic acid as the indicator.

### 9.2 Reagents

9.2.1 *Potassium dichromate*, standard volumetric solution,  $c(\text{K}_2\text{Cr}_2\text{O}_7) = 0.008 \text{ mol/l}$ .

9.2.1.1 Dry potassium dichromate at  $105^\circ\text{C}$  for one and a half hours. Cool in a desiccator and transfer 2.354 g to a one-litre volumetric flask carefully through a funnel using water at room temperature. Wash the funnel thoroughly and dissolve the reagent by shaking until the solution is complete. Make up to the mark and mix well.

9.2.2 *Hydrochloric acid*, 9 per cent (V/V) solution, prepared by using concentrated hydrochloric acid (relative density = 1.18).

9.2.3 *Silver reductor*, fill the column of the reductor with 18 g of silver prepared in the following manner.

9.2.3.1 Dissolve 29 g of silver nitrate in 400 ml of water, adding a few drops of dilute nitric acid. Then stir the solution vigorously with a 100 mm x 100 mm sheet of electrolytic copper, until silver has been completely precipitated. Transfer the silver, after washing with sulfuric acid, to the column with water. Agitate to remove all air bubbles and wash again with dilute sulfuric acid. Store the column filled with hydrochloric acid when not in use.

9.2.4 *Orthophosphoric acid*, 85 per cent (V/V) solution, (relative density = 1.69)

9.2.5 Sodium salt of *N*-methyl diphenyl *p*-sulfonic acid, 1 per cent, (m/V) solution.

### 9.3 Procedure

9.3.1 Pipette out 50 ml of the filtrate preserved in the determination of silica (in 6.3.5), into the silver reductor column (9.2.3) and pass it through the column collecting into a 500-ml beaker. Wash the column by passing 100 ml of hydrochloric acid (9.2.2) in five portions and 100 ml of water through the column and collect the washings as well into the same beaker. Pour each portion when the liquid reaches the level of silver. Add 10 ml of orthophosphoric acid (9.2.4) and 0.8 ml to 1.0 ml of sodium salt of *N*-methyl diphenylamine *p*-sulfonic acid (9.2.5). Titrate against potassium dichromate solution (9.2.1) until the colour changes to port-wine red.

### 9.4 Calculation

9.4.1 Total iron, per cent by mass =  $\frac{5.585 \times c \times V}{m}$

where,

*c* = concentration of potassium dichromate solution in moles per litre;

*V* = volume, in millilitres, of potassium dichromate required for the titration; and

*m* = mass, in grams, of the sample represented by the aliquot taken.

## 10 DETERMINATION OF ZIRCONIA

### 10.1 Outline of the method

10.1.1 Titanium and iron are removed from zirconium by fusion with potassium bisulfate and extraction with sulfuric acid. Zirconium which is obtained as residue is brought into solution by fusion with sodium carbonate and extracted with sulfuric acid. Silicon dioxide is separated by filtration after evaporation and dissolution of zirconia. Zirconium which is in the solution is then precipitated as zirconium pyrophosphate ( $ZrP_2O_7$ ) in acidic medium and weighed.

### 10.2 Reagents

10.2.1 Potassium bisulfate, solid.

10.2.2 Sulfuric acid, 3 per cent (V/V), 10 per cent (V/V) and 50 per cent (V/V) solutions, prepared by using concentrated sulfuric acid (relative density = 1.84).

10.2.3 Sodium carbonate, solid.

10.2.4 Hydrogen peroxide, 3 per cent (m/V) solution.

10.2.5 *Diammonium hydrogen phosphate*, 2.5 per cent (m/V) solution.

10.2.6 *Ammonium nitrate*, 5 per cent (m/V) solution.

### 10.3 Procedure

10.3.1 Weigh to the nearest 0.0001 g about 0.5 g of the sample. Transfer it to a silica crucible containing 6 g to 7 g of potassium bisulfate (10.2.1) and heat it over a low flame until potassium bisulfate is melted. Increase the temperature to dull red heat. As soon as the sample is completely decomposed, remove the crucible from heat and allow it to cool to room temperature. Transfer the crucible with its contents to a beaker and extract the contents with 100 ml of 10 per cent sulfuric acid (10.2.2). Warm the solution and filter through a No. 40 Whatman filter paper. Remove any adhering particles from the beaker by means of 3 per cent sulfuric acid (10.2.2). Wash the precipitate and filter paper with hot water until free from sulfate (test with barium chloride). Discard the filtrate.

10.3.2 Transfer the filter paper with the residue to a platinum crucible, ignite and fuse it with 2 g of sodium carbonate (10.2.3). Extract the melt with about 25 ml of 50 per cent sulfuric acid (10.2.2) in a basin, add 10 ml of hydrogen peroxide (10.2.4) and evaporate to dense white fumes. Continue the fuming for 30 minutes. Cool and dilute to 50 ml with hot water and filter through a No. 40 Whatman filter paper. Wash with hot water until free from sulfate (test with barium chloride). Discard the residue or if required, proceed with 6.3.4 to determine silica as a check. Collect the filtrate and washings in a 600-ml beaker. Dilute the filtrate and the washings with water so that the final volume after adding all the reagents will be 300 ml. Add 10 ml of hydrogen peroxide and 60 ml of 50 per cent sulfuric acid. Then add 25 ml of diammonium hydrogen phosphate (10.2.5) slowly while stirring. Allow to stand on a steam bath for two hours. Then leave it overnight. Filter through a No. 40 Whatman filter paper and wash with ammonium nitrate solution (10.2.6) until free from sulfate (test with barium chloride).

10.3.3 Transfer the filter paper and the precipitate to a weighed platinum crucible and heat very carefully so that the paper does not catch fire. When the paper has charred gradually, increase the temperature until all carbon is removed and then heat at about 105 °C until the difference between two successive weighings is not more than 0.0001 g. Cool in a desiccator and weigh as zirconium pyrophosphate ( $\text{ZrP}_2\text{O}_7$ ).

### 10.4 Calculation

$$\text{Zirconia per cent by mass} = \frac{m_1 \times 46.47}{m_0}$$

where,

$m_0$  = mass, in grams, of the sample; and

$m_1$  = mass, in grams of  $\text{ZrP}_2\text{O}_7$  obtained under 10.3.3.

## 11 DETERMINATION OF ALUMINA

## 11.1 Outline of the method

11.1.1 Aluminium is precipitated as oxinate from a buffered acetate solution and dissolved in hydrochloric acid followed by bromometric titration of the liberated 8-hydroxyquinoline.

## 11.2 Reagents

11.2.1 Concentrated nitric acid, relative density = 1.42.

11.2.2 Ammonium chloride, solid.

11.2.3 Concentrated hydrochloric acid, relative density = 1.18

11.2.4 Hydroxylammonium chloride, 10 per cent (m/V) solution.

11.2.5 *o*-phenanthroline monohydrate, 1 per cent (m/V) solution.

Dissolve 1 g of *o*-phenanthroline monohydrate in 90 ml of water with gentle heating and constant stirring. Cool and dilute to 100 ml.

11.2.6 Bromocresol purple indicator, 0.04 per cent (m/V) solution. Dissolve 0.04 g of bromocresol purple indicator in 100 ml of hot water. Cool and filter.

11.2.7 Ammonium hydroxide, 25 per cent (V/V) solution, prepared by using concentrated ammonium hydroxide (relative density = 0.88).

11.2.8 8-hydroxyquinoline (*Oxine*), 2 per cent (m/V) solution. Dissolve 5 g of 8-hydroxyquinoline in 15 ml of glacial acetic acid by warming and dilute with water to 250 ml.

11.2.9 Ammonium acetate, solid.

11.2.10 Potassium bromide, solid.

11.2.11 Potassium bromate, standard volumetric solution,  $c(\text{KBrO}_3) = 0.001 \text{ mol/l}$ .

Dry some finely powdered potassium bromate for one to two hours at 120 °C and allow to cool in a desiccator. Weigh to the nearest 0.0001 g about 1.392 g of the potassium bromate and dissolve it in water in a one-litre volumetric flask and bring up to the mark.

11.2.12 Potassium iodide, 10 per cent (m/V) solution.

11.2.13 Sodium thiosulfate, standard volumetric solution,  $c(\text{Na}_2\text{S}_2\text{O}_3) = \text{approximately } 0.025 \text{ mol/l}$ .

Weigh about 12.5 g of sodium thiosulfate crystals, dissolve in freshly boiled and cooled water and make up to one litre in a volumetric flask.

If the solution is to be kept for more than a few days, add 0.1 g of sodium carbonate or three drops of chloroform. Standardize this solution against potassium bromate solution (11.2.11), iodimetrically.

11.2.14 *Starch solution*, make a suspension of 1 g of soluble starch in about 5 ml of water and add it carefully to 100 ml of boiling water. Cool the solution before use. Prepare fresh as needed.

11.2.15 *Ammonium nitrate*, 5 per cent (m/V) solution.

11.2.16 *Hydrochloric acid*, 50 per cent (V/V) solution, prepared by using concentrated hydrochloric acid (relative density = 1.18)

11.2.17 *Sodium hydroxide*, solid.

### 11.3 Procedure

11.3.1 Pipette out exactly 100 ml of the solution preserved under 6.3.5 into a 500-ml beaker, add 5 ml of concentrated nitric acid (11.2.1) to oxidize iron (II) and boil. Cool and add 4 g to 5 g of ammonium chloride (11.2.2) and 5 drops of methyl red indicator. Precipitate hydroxides by first adding concentrated ammonium hydroxide and then with dilute ammonium hydroxide until the colour changes to a distinct yellow. Allow to settle, filter and wash four times with hot ammonium nitrate solution (11.2.15). Digest the precipitate of hydroxides with about 3 g of sodium hydroxide (11.2.17) for an hour on a steam-bath, dilute, filter and wash.

11.3.2 Dissolve the precipitate in hydrochloric acid (11.2.16) and repeat the digestion of the residue and filter. Add this filtrate to the filtrate in 11.3.1. Concentrate the solution and acidify with concentrated hydrochloric acid (11.2.3). Again concentrate the solution to about 150-ml. Add 2 ml of hydroxylammonium chloride (11.2.4) followed by 2 ml of *o*-phenanthroline monohydrate solution (11.2.5). Cool, add three to four drops of bromocresol purple indicator (11.2.6) and dilute with ammonium hydroxide (11.2.7) until the colour of the solution just turns to violet. Gradually warm the solution and when it attains a temperature of 60 °C to 70 °C add 25 ml to 30 ml of oxine solution (11.2.8) and 7 g to 8 g of ammonium acetate (11.2.9) slowly while stirring. Maintain the solution at 60 °C to 70 °C until the precipitate is coagulated. Filter through a sintered glass crucible of porosity No. 4 and wash the precipitate with hot water 10 to 12 times.

11.3.3 Dissolve the oxinate complex in a minimum quantity of warm concentrated hydrochloric acid and collect the solution in a 500-ml conical flask. Wash the crucible with hot water till acid free. Collect the washings also in the same conical flask. Cool the solution, add 2 g to 3 g of potassium bromide (11.2.10) and shake till the solid dissolves. Now add exactly 20 ml of potassium bromate solution (11.2.11) and keep for about 10 minutes for complete bromination. Add 10 ml of potassium iodide solution (11.2.12) and dilute to 200 ml. Titrate the liberated iodine with sodium thiosulfate, standard volumetric solution (11.2.13) using starch (11.2.14) as the indicator.

11.3.4 Pipette out exactly 20 ml of potassium bromate solution separately into a 500-ml conical flask. Add 2 g to 3 g of potassium bromide and shake until the solid dissolves. Add 10 ml of potassium iodide solution followed by about 5 ml of concentrated hydrochloric acid. Dilute the contents to 200 ml and titrate the liberated iodine against sodium thiosulfate solution using starch as the indicator.

#### 11.4 Calculation

$$(V_1 - V_2) \times c \times 0.425$$

$$\text{Alumina, per cent by mass} = \frac{\quad}{m}$$

where,

$V_1$  = volume, in millilitres, of the sodium thiosulfate solution required in 11.3.3;

$V_2$  = volume, in millilitres, of the sodium thiosulfate solution required for the unreacted bromate solution in 11.3.4 ;

$c$  = concentration of the sodium thiosulfate solution in moles per litre; and

$m$  = mass, in grams, of the sample represented by the aliquot taken.

## 12 DETERMINATION OF PHOSPHOROUS PENTOXIDE

### 12.1 Outline of the method

12.1.1 The phosphates in the sample is dissolved by sulfuric acid. Yellow phosphomolybdate formed by the reaction of ammonium molybdate is leached by butanol and reduced by stannous chloride. The transmittance is measured at 725 nm.

### 12.2 Reagents

12.2.1 *Sulfuric acid*, 5.5 per cent (V/V) and 50 per cent (V/V) solution prepared by using concentrated sulfuric acid (relative density = 1.84)

12.2.2 *Sulfuric acid, concentrated*, relative density = 1.84.

12.2.3 *Sodium hydroxide*, standard volumetric solution,  $c$  (NaOH) = 2 mol/l.

12.2.4 *Hydroxylammonium chloride*, 10 per cent (m/V) solution.

12.2.5 *Sodium fluoride*, solid.

12.2.6 *Ammonium molybdate*, 5 per cent (m/V) solution.

Dissolve 25 g of ammonium molybdate in water, add 75 ml of concentrated sulfuric acid and dilute to 500 ml.

12.2.7 *iso-butanol*.

12.2.8 *Stannous chloride*, standard solution.

12.2.8.1 Dissolve 22.5 g of stannous chloride in 5 per cent (V/V) hydrochloric acid and dilute to 50 ml with the same acid solution. This is the standard stock solution.

12.2.8.2 Dilute 0.5 ml of this stock solution to 100 ml by adding 50 ml of 5.5 per cent (V/V) sulfuric acid and water.

12.2.9 *Phosphorous*, standard solution

12.2.9.1 Dissolve 219.7 g of potassium dihydrogen phosphate ( $\text{KH}_2\text{PO}_4$ ) in 1 000 ml of water. This is the standard stock solution.

12.2.9.2 Dilute 10 ml of this stock solution to 100 ml with water. This is the working solution.

12.3 Procedure

12.3.1 Digest 2 g of the sample with 20 ml of concentrated sulfuric acid (12.2.2) in a 250-ml beaker by heating until the whole sample is disintegrated, or thick copious fumes appear. Cool with ice cubes. Filter through No. 41 Whatman filter paper and wash with ice cold water. Collect the washings and the filtrate in a 250-ml volumetric flask and make upto the mark. Discard the residue. Pipette 25 ml of the solution into a 100-ml separating funnel and another aliquot of 25 ml into a 250-ml beaker. Add a few drops of methyl red indicator into the beaker and titrate with the sodium hydroxide (12.2.3) until the colour changes from red to yellow. Add 5 ml of 5.5 per cent (V/V) sulfuric acid (12.2.1) and an amount of sodium hydroxide as indicated by the titration into the separating funnel. Dissolve any precipitate, add 1 ml of hydroxylammonium chloride solution (12.2.4) and 100 mg of sodium fluoride (12.2.5) and dilute to 50 ml.

12.3.2 Pipette 10 ml of ammonium molybdate solution (12.2.6) into the separating funnel and mix well. Pipette 20 ml of iso-butanol (12.2.7) into the solution and shake for 30 seconds. Let it stand for 1 minute, separate out the lower water layer and discard. Add 15 ml of stannous chloride solution (12.2.8) and shake for 30 seconds. Discard the water layer again. Collect the organic layer into an optical cell. Measure transmittance against iso-butanol as blank at 725 nm in a spectrophotometer exactly 5 minutes after shaking.

12.3.3 To a series of 100-ml separating funnels, pipette 0.5 ml, 1.0 ml, 1.5 ml, 2.0 ml, 2.5 ml, 3.0 ml, 3.5 ml, 4.0 ml, 4.5 ml and 5.0 ml of phosphorous standard working solution (12.2.9) and develop the colour using the same procedure and using the same amount of all reagents.

Plot transmittance values against phosphorous pentoxide concentrations to obtain a calibration graph.



## 12.4 Calculation

$$12.4.1 \text{ Phosphorous pentoxide, per cent by mass} = \frac{m_1}{m_0} \times 10^{-4}$$

where,

$m_0$  = mass, in grams, of the sample represented by the aliquot taken; and

$m_1$  = mass, in micrograms, of phosphorous pentoxide obtained from the graph.

## 13 DETERMINATION OF CHROMIUM OXIDE

### 13.1 Outline of the method

13.1.1 Chromium and vanadium are converted to chromate and vanadate by fusion with sodium carbonate and are leached out with water. Vanadium is removed from chromium in chloroform layer as vanadium hydroxyquinonate complex. Then colour is developed by complexing the chromium with diphenyl carbazide in sulfuric acid. The transmittance is measured at 540 nm.

### 13.2 Reagents

13.2.1 *Sodium carbonate*, solid

13.2.2 *Sodium carbonate*, 2 per cent (m/V) solution.

13.2.3 *Ethanol*

13.2.4 *Sulfuric acid*, standard volumetric solution,  $c(\text{H}_2\text{SO}_4) = 1 \text{ mol/l}$ .

13.2.5 *8-hydroxyquinoline*, 2.5 per cent (m/V) solution.

Dissolve 2.5 g of 8-hydroxyquinoline in 100 ml of 120 g/l acetic acid solution and store in an amber colour stoppered bottle.

13.2.6 *Chloroform*

13.2.7 *Diphenyl carbazide*, 0.25 per cent (m/V) solution.

Dissolve 0.5 g of diphenyl carbazide in 200 ml of acetone and store in an amber colour stoppered bottle.

13.2.8 *Chromium*, standard solution.

13.2.8.1 Dissolve 0.2 g of dry potassium dichromate in water in a 500-ml volumetric flask and make upto the mark. This is the standard stock solution.

13.2.8.2 Pipette 20 ml of this stock solution into a 500-ml volumetric flask and dilute to the mark with water. This is the standard working solution. 1 ml of this solution contains 0.01 mg of chromium oxide.

### 13.3 Procedure

13.3.1 Fuse 0.2 g of the sample with about 2 g of sodium carbonate (13.2.1) in a platinum crucible for 30 minutes. Then extract with a minimum amount of hot water. Filter the solution into a 100-ml volumetric flask through a No. 1 filter paper. Wash the filter paper with sodium carbonate solution (13.2.2). Collect the washings into the same flask and make up to the mark. Pipette 10 ml of the solution into a beaker and titrate against sulfuric acid (13.2.4) with methyl red as indicator until the colour changes to red. Pipette 10 ml of the solution into a 100-ml separating funnel, add an amount of sulfuric acid (13.2.4) required to neutralize as determined above and swirl to liberate carbon dioxide. Then add 0.1 ml of 8-hydroxyquinoline solution (13.2.5). Add 3 ml of chloroform and shake for one minute. Remove the chloroform layer and discard. Repeat this process twice. Collect the aqueous layer into a 100-ml volumetric flask.

13.3.2 Add 10 ml of sulfuric acid and 1 ml of the diphenyl carbazide solution (13.2.7) to the flask. Make the solution upto the mark and measure transmittance at 540 nm against reagent blank (13.3.3).

13.3.3 Prepare reagent blank by following the same procedure and adding the same amounts of reagents but without the sample.

13.3.4 To a series of 100-ml separating funnels, pipette 0.5 ml, 1.0 ml, 1.5 ml, 2.0 ml, 2.5 ml, 3.0 ml, 3.5 ml, 4.0 ml, 4.5 ml and 5.0 ml of chromium standard working solution (13.2.8) and develop the colour using the same procedure and using the same amounts of all reagents. Plot transmittance values against chromium oxide concentrations to obtain a calibration graph.

### 13.4 Calculation

$$\text{Chromium oxide, per cent by mass} = \frac{m_1}{10 m_0}$$

where,

$m_0$  = mass, in grams, of the sample represented by the aliquot taken; and

$m_1$  = mass, in milligrams, of chromium oxide as measured from the graph.

## 14 DETERMINATION OF VANADIUM PENTOXIDE

### 14.1 Outline of the method

Vanadium is brought into solution as vanadate and complexed with phosphoric acid and sodium tungstate to form a yellow coloured vanadium tungstophosphate. The colour developed is measured at 410 nm.

## 14.2 Reagents

14.2.1 *Sulfuric acid*, standard volumetric solution,  $c(\text{H}_2\text{SO}_4) = 1 \text{ mol/l}$ .

14.2.2 *Sulfuric acid*, 20 per cent (V/V) solution, prepared by using concentrated sulfuric acid (relative density = 1.84)

14.2.3 *Ortho phosphoric acid*, 85 per cent (V/V) solution, (relative density = 1.69)

14.2.4 *Sodium tungstate*, 2.5 per cent (m/V) solution.

14.2.5 *Vanadium*, standard solution

14.2.5.1 Dissolve 0.32 g of dry ammonium metavanadate in 500 ml of water. This is the stock solution

14.2.5.2 Pipette 50 ml of this solution into a 500-ml volumetric flask and make up to the mark. This is the working solution.

## 14.3 Procedure

14.3.1 Pipette 50 ml of the solution preserved under 13.3.1 into a 100-ml volumetric flask. Add an amount of sulfuric acid (14.2.1) required to neutralize the solution. (This is equal to five times the amount determined in 13.3.1). Add 2.5 ml of sulfuric acid (14.2.2), 0.8 ml of ortho phosphoric acid (14.2.3) and 1.1 ml of sodium tungstate solution (14.2.4) to the flask. Make up the volume to the mark and measure the transmittance at 410 nm against reagent blank (14.3.2).

14.3.2 Prepare the reagent blank following the same procedure and adding equal amounts of all reagents but not the sample.

14.3.3 To a series of 100-ml volumetric flasks, pipette 0.5 ml, 1.0 ml, 1.5 ml, 2.0 ml, 2.5 ml, 3.0 ml, 3.5 ml, 4.0 ml, 4.5 ml, and 5.0 ml, of vanadium standard working solution (14.2.5) and develop the colour using the same procedure and adding the same amounts of all reagents.

## 14.4 Calculation

14.4.1 Vanadium pentoxide, per cent by mass = 
$$\frac{m_1}{10 m_0}$$

where,

$m_0$  = mass, in grams, of the sample represented by the aliquot taken; and

$m_1$  = mass, in milligrams of vanadium pentoxide as measured from the graph.

## 15 DETERMINATION OF MOISTURE CONTENT

### 15.1 Outline of the method

15.1.1 Drying a sample at  $103^{\circ}\text{C}$  in a temperature controlled oven at atmospheric pressure until practically constant mass is reached.

### 15.2 Apparatus

15.2.1 *Analytical balance*, accurate to 0.001 g.

15.2.2 *Metal vessel*, with flat bottom and provided with a well fitting lid and having dimensions of approximately 70-mm diameter and 30-mm height,

15.2.3 *Temperature-controlled, electrically heated oven*, with good natural ventilation regulated so that the temperature of the air and of the shelves, in the vicinity of the sample lies between  $101^{\circ}\text{C}$  and  $105^{\circ}\text{C}$  in normal operation.

15.2.4 *Desiccator*, containing an efficient desiccant such as phosphorous pentoxide, silica gel etc. and provided with a metal plate which allows the vessel to cool rapidly.

### 15.3 Procedure

15.3.1 Weigh to the nearest 0.001 g the vessel (15.2.2) with its cover, after leaving it open for at least 30 minutes in the desiccator at room temperature. Transfer about 100 g of the material to the vessel and weigh the vessel with the cover to the nearest 0.001 g. Carry out these operations as quickly as possible to avoid any appreciable change in moisture content.

15.3.2 Place the vessel, containing the test sample, with the cover removed, in the oven (15.2.3), which has previously been set to operate at  $103 \pm 2^{\circ}\text{C}$  and close the oven. After three hours from the time when the oven attains a temperature of  $103^{\circ}\text{C}$ , open the oven, immediately close the vessel by means of its cover and remove the vessel from the oven and place it in the desiccator. Weigh the vessel after it has cooled to room temperature, the weighing being done after a time lapse of at least 45 minutes.

15.3.3 Return the vessel, with cover removed, to the oven for a period of one hour, transfer it to the desiccator after closing it, allow it to cool and weigh as before. Repeat the process of heating, cooling and weighing until the difference between two successive weighings does not exceed 0.001 g.

## 15.4 Calculation

$$15.4.1 \text{ Moisture content, per cent by mass} = \frac{m_1 - m_2}{m_1 - m_0} \times 100$$

where,

$m_0$  = mass, in grams, of the vessel with the cover;

$m_1$  = mass, in grams, of the vessel with the cover and test portion before drying; and

$m_2$  = mass, in grams, of the vessel with the cover and test portion after drying.

## 16 DETERMINATION OF GRAIN SIZE DISTRIBUTION

### 16.1 Apparatus

#### 16.1.1 Sieves

16.1.1.1 A set of standard sieves conforming to CS 124.

#### 16.1.2 Sieve shaker

16.1.2.1 A mechanically operated sieve shaker, which imparts a rotary motion and tapping action of uniform speed to the set of sieves, shall be used. The number of taps per minute shall be between 140 and 160. The entire apparatus shall be rigidly mounted by bolting to a solid foundation preferably of concrete.

#### 16.1.3 Balance

16.1.3.1 An analytical balance with a capacity of at least 100 g and a sensitivity of 0.01 g.

### 16.2 Procedure

16.2.1 Assemble the group of sieves selected, (16.1.1) in consecutive order as to size of openings, with the coarsest sieve on top, the assembly being completed by a solid collecting pan below the bottom sieve. Place on the top sieve, about 100 g of the material weighed to the nearest 0.01 g and close this sieve with a solid cover. Securely fasten the assembly in a suitable mechanical sieve shaking device (16.1.2) and operate it for a period of 15 minutes.

16.2.2 Remove the screened fractions from the nest of sieves by removing the coarsest sieve from the nest, gently tapping its contents to one side and pouring them upon a glazed paper. Brush any material adhering to the bottom of the sieve and frame into the next finer sieve with a soft brush. Turn the sieve, so removed, upside down on the paper containing the portion that had been retained on it and tap on the sieve. Weigh the fraction of the material so obtained to the nearest

0.01 g. Repeat this process for each sieve in the nest. The sum of the masses of all the fractions shall be not less than 99 per cent of the mass of the test specimens and the difference between this sum and the mass of the test specimen shall be regarded as the loss in sieving.

### 16.3 Test report

16.3.1 Express the mass of the fractions retained on each sieve and the mass of the fraction collected in the pan as percentages of the mass of the test specimen to the nearest 0.1 per cent and report as shown in Table 1. Report any fraction that is less than 0.5 per cent of the mass of the test specimen as *trace*.

TABLE 1 - Reporting of test data

Retained on sieve of aperture size, $\mu\text{m}$	Passing sieve of aperture size, $\mu\text{m}$	Per cent by mass
425	-	
355	425	
300	355	
250	300	
212	250	
180	212	
150	180	
125	150	
106	125	
90	106	
75	90	
63	75	

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