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IS 9749 (2007): Potash feldspar and soda feldspar for glass and ceramic industry - Specification [CHD 9: Ceramicware]



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भारतीय मानक  
कांच और सिरैमिक उद्योग के लिए पोटाश फेल्डस्पार एवं  
सोडा फेल्डस्पार — विशिष्टि  
( पहला पुनरीक्षण )

*Indian Standard*

POTASH FELDSPAR AND SODA FELDSPAR FOR GLASS  
AND CERAMIC INDUSTRY — SPECIFICATION

*( First Revision )*

ICS 73.060.01; 81.040.10; 81.060.10

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**BUREAU OF INDIAN STANDARDS**  
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NEW DELHI 110002

## FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Ceramicware Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1981 as 'Specification for potash feldspar for glass and pottery' to cover the requirements of potash feldspar for glass and ceramic industry. But as soda feldspar is also used in the industry, the Committee responsible for the formulation of this standard decided to revise this standard incorporating soda feldspar to keep it in line with the present industrial practice.

In this revision, the soda feldspar has been included as a new grade of feldspar. A new requirement, titanium oxide has also been included.

Feldspar is one of the most common minerals used in the ceramic and glass industries. It is an alkali-bearing aluminosilicate corresponding to the empirical formula  $R_2O, Al_2O_3, 6SiO_2$ , where R represents an alkali metal. As none of the feldspar group of minerals is found pure, potash feldspar always contains some soda feldspar and *vice-versa* with varying amounts of free quartz, iron oxide and traces of lime and magnesia resulting in a range of colours.

Feldspar is one of the most essential raw material for the whiteware industry where it is used as the universal flux in all types of ceramic bodies. Its role in glazes is also similar to that in ceramic bodies but it is to be ground finely, preferably to 75 microns, in order to ensure more uniform and thorough reaction with other ingredients.

In glass, feldspar is used for producing opal and alabaster ware and has also been widely adopted as an important source of alumina which also helps in reducing quantity of soda ash in the batch. Addition of feldspar to glass batch results in increase in chemical durability and resistance of glassware to scratch, impact and thermal shocks.

The composition of the Committee responsible for formulation of this standard is given in Annex G.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

## *Indian Standard*

# POTASH FELDSPAR AND SODA FELDSPAR FOR GLASS AND CERAMIC INDUSTRY — SPECIFICATION

( *First Revision* )

### 1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for soda and potash feldspar for glass and ceramic industry.

### 2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
264:2005	Nitric acid — Specification ( <i>third revision</i> )
265:1993	Hydrochloric acid — Specification ( <i>fourth revision</i> )
915:2006	One-mark volumetric flasks — Specification ( <i>second revision</i> )
1070:1992	Reagent grade water — Specification ( <i>third revision</i> )
1997:1982	Specification for burettes ( <i>second revision</i> )
2109:1982	Methods of sampling dolomite, limestone and other allied materials ( <i>first revision</i> )
2781:2004	Glossary of terms relating to ceramic ware ( <i>second revision</i> )

### 3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 2781 shall apply.

### 4 GRADES

4.1 There shall be four grades of the material as follows:

- a) *Grade 1* — Potash feldspar suitable for glass industry,
- b) *Grade 2* — Potash feldspar suitable for whiteware industry,

- c) *Grade 3* — Potash feldspar suitable for pottery industry other than whiteware, and
- d) *Grade 4* — Soda feldspar.

### 5 REQUIREMENTS

#### 5.1 Description

The material shall be in the form of powder/ grains prepared from natural feldspar rock. It shall be free from foreign material.

#### 5.2 Moisture Content

Unless otherwise agreed to between the purchaser and the supplier, moisture content in feldspar powder, as received, shall not exceed two percent by mass, when determined by the method prescribed in Annex A.

#### 5.3 Size Grading

5.3.1 The grain size of Grade 1 material, when tested in accordance with the method prescribed in Annex B, shall be within the following limits:

- |   |      |
|---|------|
| a) Material retained on 500-micron IS sieve,  | : 5  |
| percent by mass, <i>Max</i>   |      |
| b) Material passing through 500-micron IS sieve, but retained in 150-micron IS sieve, percent by mass, <i>Min</i> | : 80 |
| c) Material passing through 150-micron IS sieve, percent by mass, <i>Max</i>                                      | : 10 |

5.3.2 The grain size of Grade 2, Grade 3 and Grade 4 material shall be as agreed to between the purchaser and the supplier when tested as per the method prescribed in Annex B.

#### 5.4 True Specific Gravity

The true specific gravity of the material shall be between 2.5 and 2.7 when tested according to the procedure prescribed in Annex C.

#### 5.5 Reaction with Water

5.5.1 The reaction of potash and soda feldspar with water,

when tested as the difference of *pH* of the extracts, in accordance with the method prescribed in Annex D, shall not exceed the value of 2 for Grade 1 and 2 and shall not exceed the value of 2.5 for Grade 3 and 4.

### 5.6 Limit of Sodium (Applicable to Grades 1, 2 and 3 only)

Sodium salts in the extracts, when determined in accordance with D-2 shall not exceed 50 ppm (as Na<sup>+</sup>) for Grade 1 and Grade 2 and 80 ppm (as Na<sup>+</sup>) for Grade 3.

### 5.7 Fusibility

Because of the inherent sluggish fusion characteristics of feldspar, it is necessary to express the fusibility of feldspar in terms of Orton Standard Pyrometric Cone Equivalent (PCE).

5.7.1 The PCE ranges of different grades of soda and potash feldspar shall be within the following limits:

- Grade 1 — Cone 8 to 10 (1 225° to 1 260°C)
- Grade 2 — Cone 8 to 10 (1 225° to 1 260°C)
- Grade 3 — Cone 5 to 10 (1 180° to 1 260°C)
- Grade 4 — Cone 1 to 5 (1 110° to 1 160°C)

Determination of fusion point shall be carried out as prescribed in Annex E.

### 5.7.2 Fired Colour

The colour of fired feldspar shall be glassy white and it shall be free from specks.

### 5.8 Chemical Composition

Potash and soda feldspar shall also comply with the requirements prescribed in Table 1 when tested in accordance with the method prescribed in Annex F. Reference to the relevant clauses of Annex F is given in col 7 of Table 1.

## 6 PACKING AND MARKING

### 6.1 Packing

The material shall be packed in jute bags or as agreed to between the purchaser and the supplier.

### 6.2 Marking

The bags shall be marked with the following:

- Indication of source of manufacture;
- Grade of the material;
- Net mass of contents;
- Grain size; and
- Lot number or batch number to enable the material to be traced back from records.

**Table 1 Requirements of Potash and Soda Feldspar for Glass and Ceramics**  
(Clause 5.8)

Sl No.	Characteristic	Requirement				Method of Test, Ref to
		Grade 1	Grade 2	Grade 3	Grade 4	
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Loss on ignition, percent by mass, <i>Max</i>	0.6	0.6	0.8	1.0	F-3
ii)	Silica (as SiO <sub>2</sub> ), percent by mass, <i>Max</i>	67	67	68	68	F-4
iii)	Alumina (as Al <sub>2</sub> O <sub>3</sub> ), percent by mass	17–20	17–21	17–21	19–22	F-5
iv)	Ratio of silica to alumina	3.4–3.6	3.4–3.6	3.5–3.8	—	F-4 and F-5
v)	Iron oxide (as Fe <sub>2</sub> O <sub>3</sub> ), percent by mass, <i>Max</i>	0.20	0.35	0.50	0.20	F-6
vi)	Calcium and magnesium oxides (as CaO + MgO), percent by mass, <i>Max</i>	0.75	1.0	1.0	4.5	F-7
vii)	Alkalis (as K <sub>2</sub> O), percent by mass, <i>Min</i>	10.0	9.0	7.0	—	F-8
viii)	Alkalis (as K <sub>2</sub> O), percent by mass, <i>Max</i>	—	—	—	3.0	F-8
ix)	Alkalis (as Na <sub>2</sub> O), percent by mass, <i>Max</i>	4.0	4.0	6.0	—	F-8
x)	Alkalis (as Na <sub>2</sub> O), percent by mass, <i>Min</i>	—	—	—	8.0	F-8

**6.2.1 BIS Certification Marking**

Each package may also be marked with the Standard Mark.

**6.2.1.1** The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

**7 SAMPLING****7.1 Laboratory Sample**

For the purpose of testing, a truly representative and

homogeneous sample is essential. This shall be drawn and prepared in accordance with IS 2109.

**7.2 Test Sample**

Grind 5 to 10 g of the laboratory sample so that it passes through 75-micron IS Sieve. Dry to a constant mass at  $105 \pm 2^\circ\text{C}$  and use it for the purpose of chemical analysis.

**7.3 Criteria for Conformity**

The test sample shall meet all the requirements of the standard.

**ANNEX A**

(Clause 5.2)

**DETERMINATION OF MOISTURE CONTENT****A-1 PROCEDURE**

**A-1.1** Weigh accurately, nearest to 0.01 g, about 20 g of the sample in a tared platinum dish and dry at  $105^\circ$  to  $110^\circ\text{C}$  in an air-oven for 30 min. Cool in a desiccator and weigh. Repeat heating and cooling till constant mass is obtained.

**A-1.2 Calculation**

$$\text{Moisture content} = \frac{m - m_1}{m} \times 100$$

where

$m$  = mass of the sample taken for the test, in g; and  
 $m_1$  = mass of the sample after drying, in g.

**ANNEX B**

(Clauses 5.3.1 and 5.3.2)

**METHOD OF SIZE GRADING****B-1 PREPARATION OF TEST SAMPLE**

**B-1.1** Transfer about 200 g of the sample to a porcelain dish, dry to constant mass at  $105^\circ$  to  $110^\circ\text{C}$  in an air-oven and cool in a desiccator.

**B-1.2** Assemble the 500-micron and 150-micron IS Sieves, with the former on top and a pan at the bottom. Weigh accurately 100 g of the sample, nearest to 0.1 g and place it on the top sieve. Close the nest of sieves with a cover and shake the sieves in a sieve shaker for atleast 15 min. Stop

the sieve shaker, remove the sieves and weigh separately to the nearest 0.1 g, the fractions retained on each sieve. Express the results as percentages of the mass of the sample taken for the test.

NOTE — Calculate percentage of the material passing through 150-micron IS Sieve by deducting from 100 the percentages of the material retained on the two sieves.

**B-1.3** Carry out the test in duplicate and report the average of the test results on each sieve.



## ANNEX C

### (Clause 5.4)

#### DETERMINATION OF TRUE SPECIFIC GRAVITY

##### C-1 APPARATUS

**C-1.1 Analytical Balance** — accurate up to 0.000 1 g.

**C-1.2 Pyknometer** — 50 ml, with a ground capillary stopper.

**C-1.3 Vacuum Source** — to produce a vacuum of 12 to 25 mm of mercury.

##### C-2 PROCEDURE

**C-2.1** Dry the pyknometer at 105° to 110°C, cool in a desiccator and weigh, recording the mass as  $m$ . Fill the pyknometer with distilled water at room temperature  $t^\circ\text{C}$ , which should be between 15.6°C and 23.9°C and should not vary more than 0.3°C during the test. Insert the stopper and wipe the excess water from the tip in such a manner as not to withdraw any water from the capillary. Thoroughly dry any water adhering to the outside of the pyknometer and weigh recording the mass as  $m_1$ .

**C-2.2** Empty the pyknometer and dry again at 105° to 110°C. Introduce about 10 g of the dried sample (*see B-1.1*) and weigh with the stopper in position, recording the mass as  $m_2$ . Remove the stopper, fill the pyknometer with distilled water to one-fourth to one-half its capacity and boil the water for 15 min over a sand bath at atmospheric pressure or under reduced pressure, taking care that boiling is not so vigorous as to cause loss of sample due to popping.

NOTE — Where boiling is done at atmospheric pressure, it is advisable to insert a thin strip of paper with the stopper before the boiling operation.

**C-2.3** After boiling fill the pyknometer with distilled water, cool to room temperature  $t^\circ\text{C}$  in a water bath. Insert the stopper, wipe the excess water from the tip and the outside of the pyknometer as above and weigh, recording the mass as  $m_3$ .

##### C-3 CALCULATION

**C-3.1** Calculate the true specific gravity as follows:

$$\text{True specific gravity, } t^\circ/t^\circ\text{C} = \frac{(m_2 - m)}{(m_2 - m) - (m_3 - m_1)}$$

where

$t$  = temperature of the water, in °C;

$m$  = mass of empty stoppered pyknometer, in g;

$m_1$  = mass of stoppered pyknometer filled with distilled water, in g;

$m_2$  = mass of stoppered pyknometer and sample, in g; and

$m_3$  = mass of stoppered pyknometer with sample and distilled water, in g.

**C-3.2** Carry out the test in duplicate and report the result nearest to 0.01. The results shall agree to within 0.03, or additional determinations shall be made in duplicate.

## ANNEX D

(Clauses 5.5.1 and 5.6)

#### DETERMINATION OF REACTION WITH WATER

##### D-1 APPARATUS

**D-1.1 Balance** — accurate to 0.01 g.

**D-1.2 Agate Pestle and Mortar**

**D-1.3 pH Meter** — with minimum accuracy of 0.02 pH unit and one each of H<sup>+</sup> ion and Na<sup>+</sup> ion selective glass electrodes and a calomel reference electrode.

**D-1.4 Flask Shaker**

**D-1.5 Centrifuge**

##### D-2 PROCEDURE

**D-2.1 Determination of Difference in pH**

Take about 100 g of the dry sample (*see B-1.1*) and grind it to pass entirely through 75-micron IS Sieve.

**D-2.1.1** Take 50 g of powdered material in a flask and add 250 ml of distilled water. Take 250 ml of distilled water in

another flask as the blank. Allow the two flasks to stand without stirring for 20 h. Then shake both the flasks in a flask shaker for one hour. Allow to stand for 15 min. Centrifuge the contents of the two flasks and measure  $pH$  of the sample extract and the blank with the help of  $pH$  meter and glass electrode combination.

**D-2.1.2** Record the  $pH$  values obtained above and report the result as given below:

$pH$  difference =  $pH$  of the sample extract –  $pH$  of the blank

### D-2.2 Estimation of Sodium Salts in ppmNa

For characterization of soluble salts, take 25 ml of the sample extract obtained in **D-2.1.1** in a 50 ml volumetric flask, add 10 to 12 drops of triethanolamine and make up the volume with distilled water. Similarly, take 25 ml of the blank extract obtained in **D-2.1.1** and make up as above in another 50 ml volumetric flask.

**D-2.2.1** Standardize the  $pH$  meter against 0.01 N sodium chloride solution taking its  $pNa$  as 2.00 and measure the  $pNa$  (that is, the inverse logarithm of  $Na^+$  ion concentration) with the help of the  $pH$  meter using  $Na^+$  ion selective glass electrode alongwith the reference electrode.

**D-2.2.2** Calculate and report the result as given below:

$$pNa = \frac{N}{\text{antilog}(pNa)} = \frac{22.99 \times 10^3}{\text{antilog}(pNa)} = \text{ppmNa}$$

ppmNa of extracts (as in **D-2.1**)

$$= \text{ppmNa of test solutions} \times \frac{50}{25} \text{ (as in D-2.2)}$$

ppmNa soluble = ppmNa (of sample extract) – ppmNa (of blank)

## ANNEX E

(Clause 5.7.1)

### DETERMINATION OF FUSIBILITY

#### E-0 OBJECT OF TEST

The object of this test is to determine the softening point of feldspar by comparing the test cones prepared from the material under test with standard pyrometric cones heated in a suitable furnace at a definite rate of rise of temperature.

#### E-1 APPARATUS

**E-1.1 Furnace** — gas-fired; capable of maintaining a neutral or oxidizing atmosphere.

#### E-2 PREPARATION OF TEST CONES

**E-2.1** Take about 200 g of the dry sample and grind to pass through 150-micron IS Sieve, avoiding excessive reduction of size. Mix thoroughly with sufficient alkali-free dextrin, glue or starch solution in water and form into test cones in a metal mould, in the shape of tetrahedron measuring 8 mm on the sides of the base and 25 mm high. Dry these cones slowly.

#### E-3 PROCEDURE

**E-3.1** Mount the test cones along with Orton Standard Pyrometric Cones covering the appropriate range (see 5.7.1), around the outer edge of a fireclay plaque so that their bases are embedded approximately 3 mm deep, and one of the faces is inclined towards the centre of the plaque at an angle of 82° with the horizontal.

**E-3.2** Carry out heating of the mounted cones in the furnace at a uniform rate of 150°C/h until a temperature of 1 000°C is reached. Then reduce this rate to 120°C/h and continue heating till the deformation takes place. Avoid reducing conditions in the furnace during heating operation. Softening of the cones is indicated by the top bending over and the tip of the cone touching the plaque surface.

**E-3.3** Report the fusibility of the material in terms of the Orton Standard Pyrometric Cone which most nearly corresponds in time of complete bending with the test cone.

## ANNEX F

### (Clause 5.8)

#### CHEMICAL ANALYSIS OF POTASH AND SODA FELDSPAR

##### F-1 SAMPLING AND PREPARATION OF SAMPLE

The material shall be sampled and prepared for chemical analysis as given in 7.1 and 7.2.

##### F-2 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (see IS 1070) shall be used.

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

##### F-3 DETERMINATION OF LOSS ON IGNITION

**F-3.1** Transfer 1 g of the test sample to a weighed platinum crucible and introduce it into a muffle furnace and slowly raise the temperature to 950° to 1 000°C. After about 30 min, remove the crucible and cool in a desiccator and weigh. Repeat ignition and cooling to obtain constant mass.

##### F-3.2 Calculation

$$\text{Loss on ignition, percent by mass} = \frac{m - m_1}{m} \times 100$$

where

$m$  = mass of the sample taken for the test, in g; and

$m_1$  = mass of the sample after ignition, in g.

##### F-4 DETERMINATION OF SILICA

###### F-4.1 Outline of the Method

The sample is decomposed by fusion with sodium carbonate and extracted with acid. Silica is then determined gravimetrically by dehydration and baking followed by hydrofluorization. The residual silica in the filtrate is determined photometrically using molybdenum blue method.

###### F 4.2 Reagents

**F-4.2.1 Sodium Carbonate** — Anhydrous.

**F-4.2.2 Dilute Hydrochloric Acid** — (1 : 1), (1 : 3) and (1 : 19) (v/v).

**F-4.2.3 Dilute Sulphuric Acid** — 1 : 1 (v/v).

**F-4.2.4 Hydrofluoric Acid** — 40 percent (m/m).

**F-4.2.5 Potassium Pyrosulphate** — Solid.

**F-4.2.6 EDTA Solution** — 0.025 M approximately.

Dissolve 9.31 g of disodium salt of ethylene-diamine tetraacetic acid, dihydrate ( $C_{19}H_{14}N_2 Na_3O_5 \cdot 2 H_2O$ ) in water and dilute to 1 litre.

**F-4.2.7 Phenolphthalein Solution** — Dissolve 1 g of phenolphthalein in 100 ml of rectified spirit.

**F-4.2.8 Dilute Ammonia Solution** — 1 : 3 (v/v).

**F-4.2.9 Ferric Ammonium Sulphate Solution** — Dissolve 40.82 g of ferric ammonium sulphate [ $NH_4Fe(SO_4)_2 \cdot 12H_2O$ ] in water containing 80 ml of dilute hydrochloric acid (1 : 1) and dilute to 500 ml in a volumetric flask.

**F-4.2.10 Ammonium Molybdate Solution** — Dissolve 100 g of ammonium molybdate in water containing a few drops of dilute ammonia, filter if necessary, and dilute to 1 litre.

**F-4.2.11 Oxalic Acid Solution** — Dissolve 100 g of oxalic acid in water and dilute to 1 litre.

**F-4.2.12 Ascorbic Acid Solution** — 10 percent (m/m). Dissolve 10 g of the acid in 100 ml of water and filter, if necessary. Preserve in an amber coloured bottle. The solution, thus preserved, could be used up to 5 days.

**F-4.2.13 Standard Silica Solution** — 0.04 mg/ml.

- a) Fuse 0.20 g of pure silica (99.8 percent) in 5 g of sodium carbonate in a platinum crucible. Dissolve in 300 ml of water, containing 8 to 10 g of sodium hydroxide, using a polythene beaker. Cool and dilute with water in a volumetric flask to 500 ml. Store in a polythene bottle.
- b) Prepare immediately before use a standard solution containing 0.04 mg/ml by diluting the above stock solution appropriately.

###### F-4.3 Procedure

###### F-4.3.1 Decomposition of the Sample

Weigh accurately about 0.5 g of the finely ground and dried (105°C to 110°C) sample into a platinum crucible. Add 3 g of sodium carbonate; after mixing intimately, cover the dish with lid and heat over a Meker burner, slowly increasing the temperature with caution till the mixture fuses. Keep at this temperature for 10 min and finally heat till a clear melt is obtained. Remove from the burner and cool. Extract the melt in a porcelain dish first with water and then with (1 : 1) dilute hydrochloric acid.

**F-4.3.1.1** Add about 30 ml of (1 : 1) dilute hydrochloric acid and 0.5 ml of dilute sulphuric acid. Cover the porcelain dish and allow to stand on a steam bath until disintegration of the melt is complete. Crush any large flakes with flattened end of a glass rod.

#### **F-4.3.2 Gravimetric Determination of Insoluble Silica**

Evaporate the solution in the porcelain dish to dryness and allow it to remain on the steam bath for another hour. Transfer the dish to an air oven maintained between 105°C to 110°C and bake for 1 h. Cool the dish and add 20 ml of (1 : 1) dilute hydrochloric acid followed by 30 ml of hot water. Stir the mixture till solution of soluble salts appear to be complete. Digest the mixture on a steam bath for 10 min without stirring. Filter through No. 42 filter paper and rinse the dish with (1 : 19) dilute hydrochloric acid, scrubbing the basin with a rubber-tipped glass rod. Wash the precipitate five times with (1 : 19) and hot dilute hydrochloric acid and then with hot water until free from chloride. Reserve the filtrate and washings for photometric determination of residual silica.

**F-4.3.2.1** Transfer both the filter paper and precipitate to a tared platinum crucible. Ignite at a low temperature until the precipitate is free from carbonaceous matter and then heat in the muffle furnace at 1 050°C to constant mass (one hour being normally sufficient). Cool in a desiccator and weigh recording the mass as  $M_1$ . Moisten the contents of the cold crucible with water, add five drops of dilute sulphuric acid and 10 ml of hydrofluoric acid. Evaporate to dryness on a hot plate or sand bath, in a fume cupboard, taking care to avoid spurting.

**F-4.3.2.2** Heat the crucible and residue, cautiously at first, over a gas flame until the disappearance of white fumes and finally for 5 min in the furnace at 1 050°C, cool in a desiccator and weigh recording the mass as  $M_2$ .

NOTE — If the residue weighs more than 10 mg, repeat the treatment with dilute sulphuric acid and hydrofluoric acid, ignite cool, and weigh (as  $M_2$ ). Then fuse any residue with 0.5 g of anhydrous sodium carbonate, dissolve in 10 ml of (1 : 4) dilute hydrochloric acid and transfer quantitatively to the filtrate and washings reserved in F-4.3.2.

#### **F-4.3.3 Photometric Determination of the Residual Silica**

Boil the filtrate and washings reserved in F-4.3.2 and reduce to about 200 ml. Transfer to a 250 ml volumetric flask and make up to the mark with water.

##### **F-4.3.3.1 Test blank solution**

Prepare a test blank solution using the same quantities of reagent and procedure as in F-4.3.1 to F-4.3.2, but without the sample and make up to 250 ml with water.

NOTE — Fusion may, however, be omitted to avoid undue attack on platinum crucible.

**F-4.3.3.2** Transfer 10 ml aliquot of the solution prepared in F-4.3.3 to a 50 ml volumetric flask. Also transfer 10 ml of each of the test blank solution prepared in F-4.3.3.1 to five 50 ml volumetric flasks. Add to the five flasks 0, 1, 2, 3 and 4 ml respectively of the standard silica solution. Then to all the flasks add 5 ml of EDTA solution, a drop of phenolphthalein solution and then dilute ammonia solution drop-wise until the colour changes to red. Then add 5 ml of ferric ammonium sulphate solution and dilute to about 30 ml with water (the pH of the solutions should be 1 to 1.5).

**F-4.3.3.3** Add 5 ml of ammonium molybdate solution and allow to stand for 10 min. Then add 5 ml of oxalic acid solution followed immediately by 5 ml of ascorbic acid solution and dilute the volume to 50 ml. Allow the solution to stand for 30 minutes for development of full colour.

**F-4.3.3.4** Starting with the blank ('0' ml) solution, transfer a suitable portion of the coloured solution one at a time to an absorption cell, record the photometric readings using the light band centred approximately at 810 nm. Prepare the calibration curve from these readings against concentration of silica in mg (as SiO<sub>2</sub>).

**F-4.3.3.5** Similarly, transfer a suitable aliquot of coloured test solution to the absorption cell and note the photometric reading for the test solution. Convert it to milligrams of silica by means of the calibration curve and calculate the silica content in grams in 250 ml of solution prepared in F-4.3.3.

#### **F-4.4 Calculation**

$$\text{Silica, percent by mass} = \frac{M_1 - M_2 + M_3}{M} \times 100$$

where

$M$  = mass of sample taken for the test in F-4.3.1, in g;

$M_1$  = mass of crucible and contents before hydrofluoric acid treatment, in g;

$M_2$  = mass of crucible and residue after hydrofluoric acid treatment, in g; and

$M_3$  = mass of silica present in 250 ml of solution prepared in F-4.3.3, in g.

### **F-5 DETERMINATION OF ALUMINA**

#### **F-5.1 Outline of the Method**

Aluminium is separated from the aliquot of solution prepared in F-4.3.3 as sodium aluminate by treatment with sodium hydroxide and is complexed quantitatively with EDTA by boiling with an excess of the reagent. The excess EDTA is back titrated with standard zinc solution at pH about 5.3 using xylenol orange indicator. The aluminium EDTA complex on boiling with ammonium fluoride liberates

EDTA which when titrated with standard zinc acetate solution gives the amount of aluminium present in the solution.

### F-5.2 Reagents

**F-5.2.1 Dilute Sulphuric Acid** — 1 (v/v). Add 100 ml of concentrated sulphuric acid (rd 1.84) cautiously with stirring to 100 ml of water and cool.

**F-5.2.2 Hydrofluoric Acid** — 40 percent (m/m).

**F-5.2.3 Sodium Hydroxide Solution** — 10 percent (m/v).

**F-5.2.4 Sodium Carbonate** — solid.

**F-5.2.5 Dilute Hydrochloric Acid** — 1 : 1 (v/v).

**F-5.2.6 EDTA Solution (0.01 M)** — Transfer 100 ml of the solution prepared in F-4.2.6 to a 250 ml volumetric flask and dilute with water up to the mark.

**F-5.2.7 Methyl Orange** — Dissolve 0.05 g of methyl orange in 100 ml of water.

**F-5.2.8 Dilute Ammonia (1 : 5)** — Dilute 25 ml of concentrated ammonia (rd 0.90) to 150 ml.

**F-5.2.9 Acetate Buffer (pH 5.3)** — Dissolve 21.5 g of sodium acetate ( $\text{CH}_3\text{COONa}$ ,  $3\text{H}_2\text{O}$ ) in 300 ml of water containing 2 ml of glacial acetic acid and dilute to 1 litre.

**F-5.2.10 Standard Aluminium Solution** — Weigh 0.5293 g of aluminium metal (99.9 percent purity) into a beaker containing 40 ml of dilute hydrochloric acid (1 : 1) and small drops of mercury. Heat the beaker over a water bath. Transfer the solution to a 1 000 ml volumetric flask and make up to the mark. One millilitre of this solution is equivalent to 1.0 mg of alumina (as  $\text{Al}_2\text{O}_3$ ).

NOTE — Mercury will not dissolve and may be removed while transferring the solution to 1 000 ml volumetric flask.

**F-5.2.11 Standard Zinc Acetate Solution (0.01 M)** — Dissolve 5.487 g of zinc acetate [ $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ ] in 50 ml of water. Add 0.5 ml of glacial acetic acid and 25 ml of acetate buffer (see F-5.2.9) and dilute to 1 litre in a volumetric flask. Further dilute 100 ml of this solution to 250 ml in a volumetric flask and find out the equivalent  $\text{Al}_2\text{O}_3$ , in g/ml, of this diluted solution from the procedure given in F-5.3.3, taking 20 ml of standard aluminium solution (see F-5.2.10).

**F-5.2.12 Xylenol Orange Indicator** — Dissolve 0.1 g of xylenol orange in 100 ml of water containing two drops of dilute hydrochloric acid (1 : 1).

**F-5.2.13 Ammonium Fluoride** — Dissolve 20 g of

ammonium fluoride in 100 ml of water. Add 3 drops of xylenol orange (see F-5.2.12) and then dilute hydrochloric acid (1 : 9) dropwise till the colour changes to yellow.

### F-5.3 Procedure

#### F-5.3.1 Solution of the Sample

Weigh accurately about 2 g of the finely ground test sample (see F-1.1) in a platinum dish. Moisten the sample with 5 ml of water. Add 3 ml of dilute sulphuric acid and 20 ml of hydrofluoric acid and evaporate slowly to dryness on the sand bath or hot plate in a fume cupboard, taking care to avoid spurting. Repeat the process with 10 ml of hydrofluoric acid. Cool, add 2 ml of dilute sulphuric acid and evaporate as before. Heat the dry residue cautiously until fumes of sulphur trioxide cease to be evolved. Raise the temperature to 1 000°C and ignite for 5 min. Cool and fuse the residue with 1 g of sodium carbonate and dissolve in hydrochloric acid, cool and dilute to 250 ml in a volumetric flask.

**F-5.3.2** Transfer 100 ml aliquot of the solution prepared in F-5.3.1 to a 500 ml beaker, and nearly neutralize with sodium hydroxide solution. Add with constant stirring another 100 ml of sodium hydroxide solution containing 1 g of sodium carbonate dissolved in it. Allow to stand on a warm water bath for 1 h. Finally cool to room temperature, transfer quantitatively to a 250 ml volumetric flask and dilute up to the mark with water. Mix well and allow to stand for a few minutes. Filter through a filter paper previously washed with sodium hydroxide solution, collecting the filtrate in a beaker, rinsing the beaker first with a little filtrate.

NOTE — Sodium carbonate is used for precipitating calcium and magnesium alongwith iron and titanium.

**F-5.3.3** Transfer a suitable aliquot from the filtrate to a 250 ml conical flask, add two drops of methyl orange indicator followed by (1 : 1) dilute hydrochloric acid drop-wise until the colour changes from yellow to red. Add sufficient EDTA solution to provide an excess of at least 5 ml over the expected amount of alumina (1 ml of 0.025 M EDTA = 1.25 mg  $\text{Al}_2\text{O}_3$  approximately). Add dilute ammonia drop-wise until the colour changes from red to yellow. Add 5 ml of acetate buffer and about 25 ml water and heat to boiling for 5 min. Cool, add 5 ml of buffer solution and titrate with standard zinc solution using 5 to 6 drops of xylenol orange indicator. The end point is indicated by a clear change from yellow to deep orange colour. Add 10 ml of ammonium fluoride solution and heat to boiling for 5 min. Cool, add 5 ml of buffer solution and titrate the liberated EDTA with standard zinc acetate solution.

#### F-5.4 Calculation

$$\text{Alumina, percent by mass} = \frac{V \times F}{M} \times 100$$

where

$V$  = volume of standard zinc required for the titration of liberated EDTA, in ml;

$F$  = equivalent  $Al_2O_3$  of standard zinc acetate solution, in g/ml; and

$M$  = mass of the sample represented by 100 ml of solution taken in F-5.3.2, in g.

## F-6 DETERMINATION OF IRON OXIDE

### F-6.1 Outline of the Method

The orange-red complex produced in acid solution of the sample (pH 4 to 6) by the addition of *o*-phenanthroline is determined photometrically at approximately 510 nm.

### F-6.2 Reagents

**F-6.2.1 Tartaric Acid Solution** — 10 percent (m/v).

**F-6.2.2 Hydroxylamine Hydrochloride Solution** — Dissolve 1 g of the reagent in water. Transfer the solution to a 100 ml volumetric flask, dilute to the mark and mix well.

**F-6.2.3 Ortho-Phenanthroline 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.

**F-6.2.4 Acetate Buffer** — Dissolve 21.5 g of sodium acetate ( $CH_3COONa, 3H_2O$ ) in 300 ml of water containing 2 ml of glacial acetic acid and dilute to 1 000 ml.

**F-6.2.5 Standard Iron Solution** — Dissolve 0.2 g of pure iron in concentrated hydrochloric acid. Dilute the solution to about 100 ml. Transfer to a 1 000 ml volumetric flask, dilute to the mark and mix well. Dilute 100 ml of this solution to 1 000 ml in a volumetric flask. One millilitre of this diluted solution is equivalent to 0.02 mg of iron (as Fe).

### F-6.3 Procedure

Transfer 5 ml aliquot of the solution prepared in F-5.3.1 to a 100 ml volumetric flask. Also transfer 0, 1, 2, 4, 6, 8 and 10 ml each of the standard iron solution to seven separate 100 ml volumetric flasks. Then add to each flask 2 ml of tartaric acid solution followed by 2 ml of hydroxylamine hydrochloride solution. Stir well and add 5 ml of *o*-phenanthroline solution and 10 ml of acetate buffer solution. Allow to stand for 15 min and dilute up to the mark with water.

**F-6.3.1** Starting with the blank (0 ml) solution transfer a suitable portion of the coloured solution, one at a time, to an absorption cell; record the photometric readings using the light band centred approximately at 510 nm, prepare the calibration curve from these readings against concentration of iron (as Fe).

**F-6.3.2** Similarly transfer a suitable aliquot of the coloured test solution to the absorption cell and note the photometric reading for the test solution. Convert it to milligrams of iron by means of the calibration curve and calculate the iron oxide content in the aliquot taken in F-6.3.

### F-6.4 Calculation

$$\text{Ferric oxide (as } Fe_2O_3\text{), percent by mass} = \frac{M}{M_1} \times 0.1429$$

where

$M$  = mass of iron (as Fe) found in aliquot, in mg; and

$M_1$  = mass of the sample represented by the aliquot taken in F-6.3, in mg.

## F-7 DETERMINATION OF CALCIUM OXIDE AND MAGNESIUM OXIDE

### F-7.1 Outline of the Method

In an aliquot of the sample solution,  $R_2O_3$  group elements are masked by triethanolamine, and calcium and magnesium are titrated with EDTA solution at pH 10 using eriochrome black T indicator. In another aliquot magnesium is precipitated out at pH 12 in presence of triethanolamine and calcium is titrated with EDTA using Patton and Reader (P & R) indicator. Subtracting the titre value of calcium from that of calcium and magnesium, the titre value for magnesium is obtained.

### F-7.2 Reagents

**F-7.2.1 Concentrated Nitric Acid** — see IS 264.

**F-7.2.2 Perchloric Acid** — 70 percent (m/m).

**F-7.2.3 Hydrofluoric Acid** — 40 percent (m/m).

**F-7.2.4 Concentrated Hydrochloric Acid** — see IS 265.

**F-7.2.5 Triethanolamine Solution** — 30 percent (v/v). Dilute 60 ml of triethanolamine to 200 ml.

**F-7.2.6 Sodium Hydroxide Solution** — 5N approximately. Dissolve 100 g of sodium hydroxide in water, cool and dilute to 500 ml and store in a polythene bottle.

**F-7.2.7 P & R Indicator** — Mix by grinding together 0.1 g of P & R indicator and 30 g of sodium chloride. About 0.2 g of the mixture is required for each titration.

**F-7.2.8 Standard EDTA Solution (0.01 M)** — Same as in F-5.2.6. Standardize against the standard calcium solution (see F-7.2.9) using P & R indicator and against the standard magnesium solution (see F-7.2.10) using 'Mixed' indicator.

**F-7.2.9 Standard Calcium Solution** — Dissolve 1.784 8 g of calcium carbonate dried at 150°C in a slight excess of hydrochloric acid, boil to expel carbon dioxide, cool and dilute to 1 000 ml. One millilitre of this solution is equivalent to 1 mg of calcium oxide (as CaO).

**F-7.2.10 Standard Magnesium Solution** — Wash a piece of magnesium in dilute hydrochloric acid to remove any oxide film, then with water and dry it with ethanol followed by ether. Dissolve 0.603 2 g of the metal in a slight excess of dilute hydrochloric acid, cool and dilute to 1 000 ml. One millilitre of this solution is equivalent to 1 mg of magnesium oxide (as MgO).

**F-7.2.11 Ammonia-Ammonium Chloride Buffer Solution** (pH = 10) — Dissolve 57.5 g of ammonium chloride in 250 ml of water, and 570 ml of ammonia (rd 0.88) and dilute to 1 litre and mix thoroughly.

**F-7.2.12 Eriochrome Black T Indicator Solution** — Dissolve 0.4 g of the sodium salt of eriochrome black T in a mixture of 20 ml of ethanol and 30 ml of triethanolamine.

### F-7.3 Procedure

#### F-7.3.1 Solution of the Sample

Weigh accurately about 1 g of the test sample (see F-1.1) in a platinum dish. Add 5 ml of concentrated nitric acid, 5 ml of perchloric acid and about 10 ml of hydrofluoric acid. Cover the dish with a lid. Allow the reaction to proceed for at least 15 min, then remove the lid and evaporate the mixture to dryness on a sand bath in a fume cupboard, taking care to avoid spurting. Cool, add 5 ml of perchloric acid, rinse the lid and sides of the dish with water and again evaporate carefully to dryness. To the cool, dry residue, add 2 ml of concentrated hydrochloric acid and about 15 ml of deionized water. Digest on a steam bath for 10 min, cool, filter if necessary and dilute the solution with deionized water to 250 ml in a volumetric flask. Store the solution in a polythene bottle.

**F-7.3.2 Pipette** 50 ml of the solution (see F-7.3.1) into a 250 ml conical flask. Add 10 ml triethanolamine solution. Add 15 ml of ammonia-ammonium chloride buffer solution. Add 4 to 5 drops of eriochrome black T indicator solution and immediately titrate with the standard EDTA solution until the colour changes from wine red to blue.

NOTE — If sharp end-point is not obtained, add 2 ml of potassium cyanide (10 percent, *m/v*) after the addition of triethanolamine.

#### F-7.3.2.1 Calculation

Magnesium oxide and calcium oxide, percent by mass

$$= \frac{V \times F}{M} \times 100$$

where

$V$  = volume of standard EDTA solution required for titration, in ml;

$F$  = MgO equivalent of the standard EDTA solution, in g/ml; and

$M$  = mass of the sample represented by the aliquot taken, in g.

## F-8 DETERMINATION OF ALKALIS AS POTASSIUM OXIDE AND SODIUM OXIDE

### F-8.1 General

Sodium and potassium contents are determined with the help of a flame emission spectrophotometer by measuring the intensity of radiation emitted at 767 nm by potassium or at 589 nm by sodium. For this purpose use may also be made of a filter type flame photometer. In both the cases, however, the intensity of radiation either at the specified wavelength, or through the relevant filter is compared with that obtained for simulated standard solutions prepared from potassium chloride and sodium chloride solutions.

NOTE — Water used in preparing standard solutions shall be double-distilled so that it produces no deflections of the galvanometer when the instrument is set for full scale deflection for 10 ppm of potassium or 5 ppm of sodium concentration.

### F-8.2 Apparatus

**F-8.2.1 Flame Photometer** — Provided with arrangement for monochromatic light or a set of filters required for determination of potassium and sodium. The photometer comprises essentially a galvanometer, a photo cell or tube and a photo-multiplier tube. Other accessories are an atomizer-burner (or an atomizer and a burner) fed with liquefied petroleum gas (LPG) and compressed air (or oxygen, acetylene, etc) in such a ratio that combustion of gas is complete.

**F-8.2.2 Burette** — of 50 ml nominal capacity and graduated in 0.1 ml for Class A accuracy (see IS 1997).

**F-8.2.3 Volumetric Flasks** — two of 1 000 ml, two of 250 ml and 14 of 100 ml nominal capacity, of Class A accuracy (see IS 915).

### F-8.3 Reagents

**F-8.3.1 Hydrochloric Acid** — 1 : 1 (*v/v*).

**F-8.3.2 Standard Sodium Solutions** — Weigh accurately 0.508 4 g of sodium chloride (NaCl), previously dried to constant mass at 110° ± 2°C. Dissolve in water, transfer to a 1 000 ml volumetric flask, dilute to the mark with water and mix.

**F-8.3.2.1** Transfer 10 ml of this solution to a 100 ml

volumetric flask, dilute up to the mark with water and mix. This diluted solution contains 200 ppm of sodium (as Na).

**F-8.3.2.2** Transfer 25, 20, 15, 10 and 5 ml respectively of the standard sodium solution prepared in **F-8.3.2.1** to five 100 ml volumetric flasks. Add 37.5 ml each of potassium solution prepared in **F-8.3.3** to the five flasks, dilute up to the mark with water and mix. These solutions contain 50, 40, 30, 20 and 10 ppm respectively of sodium (as Na).

**F-8.3.3 Standard Potassium Solutions** — Weigh accurately 0.3814 g of potassium chloride (KCl), previously dried at  $110^{\circ}\text{C} \pm 2^{\circ}\text{C}$  to constant mass. Dissolve in water, transfer to a 1000 ml volumetric flask, dilute up to the mark with water and mix.

**F-8.3.3.1** Transfer 10 ml of this solution to a 100 ml volumetric flask, dilute up to the mark with water and mix. This diluted solution contains 200 ppm of potassium (as K).

**F-8.3.3.2** Transfer 35, 30, 25, 20, 15, 10 and 5 ml respectively of the standard potassium solution prepared in **F-8.3.3.1** to seven 100 ml volumetric flasks. Add 26.0 ml each of the sodium solution prepared in **F-8.3.2**, to the seven flasks, dilute up to the mark with water and mix. One millilitre of these solutions is respectively equivalent to 70, 60, 50, 40, 30, 20 and 10 ppm of potassium (as K).

#### F-8.4 Setting-up the Apparatus

Switch the instrument on and wait for its stabilization. Adjust the slit and wavelengths (767 nm for potassium or 589 nm for sodium) or place the filters in position as the case may be.

**F-8.4.1** Pour water in the spray cup, open its tap and turn the compressed air or oxygen on. Adjust the tap and pressure to establish atomization of water in a fine spray. Open the gas tap and light the burner. Adjust gas, if necessary, to get complete combustion. Next switch the photocell on and adjust the indicator needle or the indicator light spot, as the case may be, to bring it to the 'zero' position.

**F-8.4.2** Atomize potassium (70 ppm, see **F-8.3.3.2**) or sodium (50 ppm, see **F-8.3.2.2**) solutions in place of water (see **F-8.4.1**) and adjust the deflection to full scale. Atomize water once again to check the indicator needle or light spot rests at 'zero'. If it shifts from 'zero' again adjust it to 'zero'. Thus when the instrument reads 'zero' with water and shows full scale deflection for 70 ppm K, or 50 ppm Na solution, it is ready for use.

#### NOTES

1 Atomization of any solution should always be followed by atomization of double-distilled water till the indicator comes to zero position.

2 Do not disturb the setting of the atomizer or burner so that rates of flow are constant for a series of determinations.

#### F-8.5 Procedure

##### F-8.5.1 Preparation of the Sample Solution

Transfer 50 ml of the solution prepared in **F-7.3.1** to a 500 ml volumetric flask and dilute up to the mark with water.

##### F-8.5.2 Calibration Graph and Determination

Without disturbing the setting of the instrument, atomize standard potassium (see **F-8.3.3.2**) or sodium (see **F-8.3.2.2**) solutions of different concentrations in an ascending order and plot the readings of the indicator against concentrations and draw the graph. Next atomize the sample solution reserved in **F-8.5.1** and note the deflection and calculate concentration of potassium or sodium as the case may be.

*Example:*

Suppose reading for 70 ppm K solution is 90.0, that for 60 ppm K solution is 72.0 and that for the sample solution is  $T$  (somewhere between 90.0 and 72.0)

Similarly, suppose reading for 40 ppm Na solution is 80.0, that for 50 ppm Na solution is 100.0; and that for sample solution is  $T$  (somewhere between 80.0 and 100.0)

Then concentration of potassium ( $C_K$ ) in the sample solution is expressed as:

$$C_K = 60 + \frac{70 - 60}{90 - 72} \times (T - 72) \text{ ppm, or}$$

$$C_K = 70 - \frac{70 - 60}{90 - 72} \times (90 - T) \text{ ppm}$$

Similarly, concentration of sodium ( $C_{Na}$ ) in the sample solution is expressed as:

$$C_{Na} = 40 + \frac{50 - 40}{100 - 80} \times (T - 80) \text{ ppm, or}$$

$$C_{Na} = 50 - \frac{50 - 40}{100 - 80} \times (100 - T) \text{ ppm}$$

#### F-8.6 Calculation

- Alkalis (as  $\text{K}_2\text{O}$ ), =  $1.204 C_K$ , and percent by mass
- Alkalis (as  $\text{Na}_2\text{O}$ ), =  $1.348 C_{Na}$  percent by mass

where

$C_K$  = concentration of potassium (as K) in test solution, in ppm; and

$C_{Na}$  = concentration of sodium (as Na) in test solution, in ppm.



## ANNEX G (Foreword)

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