
प्रक्षेपित बेरियम कार्बोनेट, तकनीकी —
विशिष्टि
(दूसरा पुनरीक्षण)

Precipitated Barium Carbonate,
Technical — Specification
(Second Revision)

ICS 71.060.50

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FOREWORD

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

This standard was first published in 1965 and subsequently revised in 1984. In the first revision, an additional grade of the material used in TV glass ceramics was incorporated.

In this revision, instrumental test method for the determination of iron has been added as an alternate test method. In addition to this, editorial corrections have been made wherever required. Also, reference clause and amendment no. 1 have been incorporated. Further, packing and marking clause has been updated.

Barium carbonate is one of the most important barium chemicals, excluding those which are used for pigments. It is used as a raw material in the manufacture of other barium compounds like barium chloride, barium nitrate, barium sulphate, barium hydroxide, barium oxide, etc. Barium carbonate is used in large quantities in conjunction with hydrochloric acid for removal of sulphate ions from sodium chloride (brine) used in the electrolytic cells for the manufacture of caustic soda. It is also used in the removal of sulphates (present as an impurity in small amounts) from water together with the precipitation of associated heavy metals, alkaline earth metals and magnesium ions as their corresponding carbonates. The other important uses to which it is put are as a flux in the ceramic industry, as an ingredient in the optical glasses and special fine glassware, in the treatment of steel required for the manufacture of high resistance steel articles, like ball bearings, in the manufacture of heat treatment and case hardening compounds and as an ingredient in the composition used for rat poisoning compound.

The composition of the Committee responsible for formulation of this standard is given in [Annex D](#).

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 : 2022 'Rules for rounding off numerical values (*second revision*)'. 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
**PRECIPITATED BARIUM CARBONATE, TECHNICAL—
SPECIFICATION**
(*Second Revision*)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for precipitated barium carbonate, technical.

2 REFERENCES

The standards listed in [Annex A](#) 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 Indian Standard are encouraged to investigate the possibility of applying the most recent edition of these standards.

3 GRADES

There shall be two grades of the material, namely:

- a) *Grade 1* — for use in electro-ceramics; and
- b) *Grade 2* — for use in the manufacture of barium salts, removal of sulphate ions, as a flux in the ceramic industry, glassware, high resistance steel, heat treatment compounds and as rat poisoning compound.

4 REQUIREMENTS**4.1 Description**

The material shall be in the form of white powder or friable mass.

4.2 The material shall comply with the requirements prescribed in [Table 1](#) when tested according to methods given in [Annex B](#). Reference to the relevant clauses of [Annex B](#) is given in (5) of the [Table 1](#).

5 PACKING AND MARKING**5.1 Packing**

The material shall be packed in jute bags lined with polyethylene films or HDPE bags. The bags shall be securely closed.

5.2 Marking

The bags shall bear legibly and indelibly the following information:

- a) Name and grade of the material;
- b) Name of the manufacturer and his recognized trade-mark, if any;
- c) Gross and net mass;
- d) Date of manufacture;
- e) Batch number; and
- f) Poison.

5.2.1 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in [Annex C](#).

Table 1 Requirements for Precipitated Barium Carbonate, Technical*(Clause 4.2)*

Sl No.	Characteristic	Requirement		Method of Test (Ref to Cl No. in Annex B)
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Barium (as Ba), percent by mass, <i>Min</i>	68.2	68.2	B-2
ii)	Carbonate (as CO ₃), percent by mass, <i>Min</i>	29.8	29.8	B-3
iii)	Moisture, percent by mass, <i>Max</i>	0.20	0.25	B-4
iv)	Matter insoluble in hydrochloric acid, percent by mass, <i>Max</i>	1.0	1.6	B-5
v)	Free alkali	-	To pass test	B-6
vi)	Water soluble sulphide (as BaS), percent by mass, <i>Max</i>	0.3	0.4	B-7
vii)	Total sulphur (as SO ₄), percent by mass, <i>Max</i>	0.70	-	B-8
viii)	Alkali and other metal sulphates (as BaSO ₄), percent by mass, <i>Max</i>	0.01	2.0	B-9
ix)	Sodium oxides (as Na ₂ O), percent by mass, <i>Max</i>	0.01	-	B-10
x)	Iron (as Fe ₂ O ₃), percent by mass, <i>Max</i>	0.01	0.04	B-11
xi)	Tap density, g/ml	1.0 to 1.5	-	B-12
xii)	Residue retained on 63 micron IS sieve, percent by mass, <i>Max</i>	0.1	-	B-13
xiii)	Reaction efficiency (after 45 min), percent by mass, <i>Min</i>	-	85	B-14

ANNEX A

(Clause 2)

LIST OF REFERRED STANDARDS

<i>IS No.</i>	<i>Title</i>	<i>IS No.</i>	<i>Title</i>
IS 75 : 1973	Specification for linseed oil, raw and refined (<i>second revision</i>)	IS 2316 : 1990	Methods of preparation of standard solutions for colorimetric and volumetric analysis (<i>second revision</i>)
IS 264 : 2005	Nitric acid — Specification (<i>third revision</i>)	IS 3025	Methods of sampling and test (physical and chemical) for water and wastewater
IS 265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)	(Part 2) : 2019/ ISO 11885 : 2007	Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (<i>first revision</i>)
IS 266 : 2024	Sulphuric acid — Specification (<i>fourth revision</i>)	(Part 65) : 2022/ ISO 17294-2 : 2016	Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including uranium isotopes (<i>first revision</i>)
IS 695 : 2020	Acetic acid — Specification (<i>fourth revision</i>)	IS 4161 : 2023	Nessler cylinder — Specification (<i>first revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)		
IS 2142 : 2023	Bromine, technical — Specification (<i>third revision</i>)		

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ANNEX B

(Clause 4.2 and Table 1)

METHODS OF TEST FOR BARIUM CARBONATE, TECHNICAL

B-1 QUALITY OF REAGENTS

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

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

B-2 DETERMINATION OF BARIUM

B-2.1 Outline of the Method

Barium is precipitated as barium chromate which is then dissolved in dilute hydrochloric acid and treated with solid potassium iodide. The liberated iodine is titrated against standard sodium thiosulphate solution.

B-2.2 Reagents

B-2.2.1 Dilute Hydrochloric Acid — 1 : 1 (v/v)

B-2.2.2 Concentrated Hydrochloric Acid — see IS 265

B-2.2.3 Concentrated Ammonium Hydroxide — 20 percent (m/m)

B-2.2.4 Ammonium Chloride — solid

B-2.2.5 Hydrogen Sulphide — gas

B-2.2.6 Lead Acetate Paper

B-2.2.7 Methyl Orange Indicator

Dissolve 0.1 g of methyl orange in 100 ml of water.

B-2.2.8 Glacial Acetic Acid — see IS 695

B-2.2.9 Ammonium Acetate Solution — 30 percent (m/v)

B-2.2.10 Ammonium Chromate Solution — 10 percent (m/v), neutral to methyl orange

B-2.2.11 Ammonium Chromate Solution — 1 percent (m/v), neutral to methyl orange

B-2.2.12 Ammonium Carbonate Solution — approximately 20 percent (m/v)

B-2.2.13 Silver Nitrate Solution — approximately 2 percent (m/v)

B-2.2.14 Dilute Nitric Acid — approximately 2 N

B-2.2.15 Potassium Iodide — solid

B-2.2.16 Standard Sodium Thiosulphate Solution — 0.1 N

B-2.2.17 Starch Indicator Solution

B-2.3 Procedure

Weigh accurately about 0.25 g of the material and dissolve in about 30 ml of dilute hydrochloric acid, add 100 ml of water, boil and filter. Wash the residue with water. Collect the filtrate and the washings in a 500 ml beaker and make it alkaline with concentrated ammonium hydroxide. Add about one gram of ammonium chloride, pass hydrogen sulphide gas and filter. Pass hydrogen sulphide gas through the filtrate again till no turbidity appears. Boil the filtrate to drive away hydrogen sulphide gas and test for complete removal of the gas by lead acetate paper. Neutralize the solution with dilute hydrochloric acid using methyl orange indicator. Add 5 to 6 drops of glacial acetic acid together with ammonium acetate solution to neutralize any free mineral acid. Heat the solution and add an excess of ammonium chromate solution.

B-2.3.1 Allow the precipitate of a barium chromate to settle for an hour. Filter through a filter paper and wash with one percent ammonium chromate solution until the filtrate is free of soluble strontium and calcium salts which are tested if a portion of the filtrate produces no cloudiness with ammonium hydroxide and ammonium carbonate. Finally wash the residue free of ammonium chromate with water till a portion of the filtrate gives only slight reddish brown colour with silver nitrate solution.

B-2.3.2 Pierce the filter paper containing the barium chromate precipitate and wash the precipitate into a beaker with hot dilute nitric acid. Dilute the solution to about 200 ml and boil. Add ammonium acetate solution to neutralize any free nitric acid. Heat and add sufficient ammonium chromate solution. Filter and repeat washings as in **B-2.3.1** above. Dissolve the precipitate of barium chromate finally in 50 ml to 100 ml of dilute hydrochloric acid, add about 2 g of solid potassium iodide and allow to react for ten minutes. Titrate the liberated iodine with standard sodium thiosulphate, using starch indicator.

B-2.4 Calculation

$$\text{Barium, percent by mass} = \frac{0.4579 \times V}{M}$$

where

V = volume, in ml, of standard sodium thiosulphate required for the titration; and

M = mass, in g, of the material taken for the test.

B-3 DETERMINATION OF CARBONATES

B-3.1 Outline of the Method

Carbonates are determined by absorbing the carbon dioxide evolved in weighed soda asbestos bulbs.

B-3.2 Apparatus — the assembly of the apparatus is shown in Fig. 1

B-3.3 Reagent

B-3.3.1 Dilute Hydrochloric Acid — 1 : 1 (v/v)

B-3.3.2 Concentrated Sulphuric Acid — see IS 266

B-3.3.3 Copper Sulphate — anhydrous

B-3.3.4 Anhydrous Magnesium Perchlorate

B-3.3.5 Soda Asbestos — size range 0.7 mm to 1.2 mm

B-3.4 Procedure

Weigh accurately about 0.5 g to 0.75 g of the material and transfer it to the flask and cover with water. Insert the stopper carrying the separating funnel and condenser and connect the latter with *D*, *C* and *B*. Pass air free from carbon dioxide through

the system. Close the stopcock in the separating funnel and insert the weighed bulbs *A*, and *A* in the train. Fill half the separating funnel with dilute hydrochloric acid, replace the stopper carrying the air and see that there is free passage for gases through the train. Open the stopcock in the separating funnel and run acid into the flask slowly. When effervescence diminishes start a flow of water through the condenser and heat the flask slowly so as to secure steady but quite ebullition. When all the carbon dioxide has been boiled out of the solution, remove the flame, introduce the current of air and sweep out all carbon dioxide. Disconnect the weighed bulbs, close and inlet and outlet tubes and place them in the balance case. When cool, open the stopper momentarily and weigh against a similar tube used as a counterpoise:

B-3.5 Calculation

$$\text{Carbonates, percent by mass} = \frac{136.4 \times m}{M}$$

where

m = increase in mass, in g, due to carbon dioxide absorption; and

M = mass, in g, of the material taken for the test.

B-4 DETERMINATION OF MOISTURE

B-4.1 Procedure

Weigh accurately about 5 g of the material in a clean squat type glass weighing bottle, previously dried and weighed. Place the weighing bottle in an oven maintained at $(110 \pm 2) ^\circ\text{C}$ till constant mass is obtained.

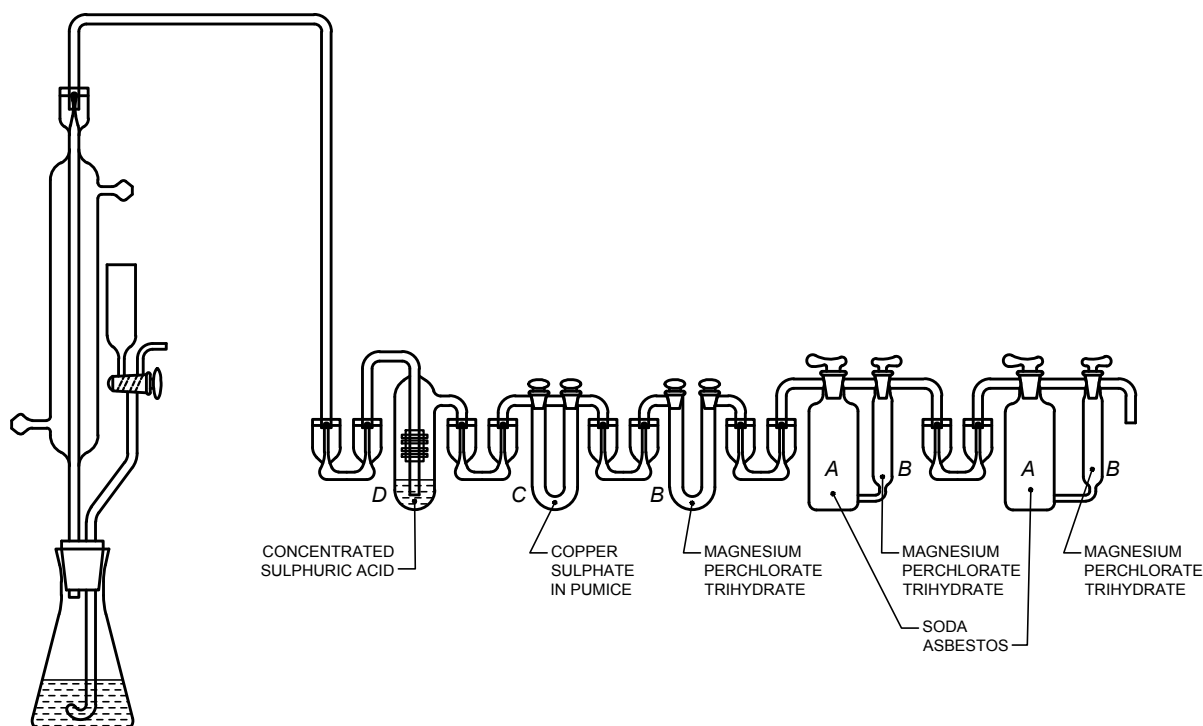


FIG. 1 APPARATUS FOR THE DETERMINATION OF CARBONATES

B-4.2 Calculation

$$\text{Moisture, percent by mass} = 100 \frac{(M_1 - M_2)}{(M_1 - M)}$$

where

M_1 = mass, in g, of the weighing bottle with the sample before drying,

M_2 = mass, in g, of the weighing bottle with the sample after drying; and

M = mass, in g, of the empty weighing bottle.

B-5 DETERMINATION OF MATTER INSOLUBLE IN HYDROCHLORIC ACID**B-5.1 Reagents**

B-5.1.1 Concentrated Hydrochloric Acid — see IS 265

B-5.2 Procedure

Mix 10 g of the material with 100 ml of water and add slowly 10 ml of concentrated hydrochloric acid. Dilute with water to 200 ml and heat on the steam-bath for 30 min. Filter the hot solution through a previously dried and weighed sintered glass crucible. Wash the residue with water slightly acidified with hydrochloric acid. Again, wash the crucible with hot water till the filtrate is free from acid. Place the crucible in an oven maintained at (110 ± 2) °C for 2 h. Cool the crucible in a desiccator and weigh.

B-5.3 Calculation

Matter insoluble in hydrochloric acid, percent by mass

$$= \frac{100 \times (M_2 - M_1)}{M}$$

where

M_2 = mass, in g, of the crucible after filtration;

M_1 = mass, in g, of the crucible before filtration; and

M = mass, in g, of the material taken for the test.

B-6 TEST FOR FREE ALKALI**B-6.1 Outline of the Method**

Free alkali is determined by titration with standard hydrochloric acid.

B-6.2 Reagents

B-6.2.1 Standard Hydrochloric Acid — 0.02 N

B-6.2.2 Phenolphthalein Indicator

Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent rectified spirit

B-6.3 Procedure

Shake 0.2 g of the material for 5 min with 30 ml of carbon dioxide-free water. Filter and add 5 drops of phenolphthalein to 20 ml of the filtrate. Titrate this solution with standard hydrochloric acid.

B-6.3.1 The material shall be taken to have passed the test, if not more than 0.25 ml of standard hydrochloric acid is required for the titration.

B-7 DETERMINATION OF WATER SOLUBLE SULPHIDES**B-7.1 Outline of the Method**

Water soluble sulphides are determined iodimetrically

B-7.2 Reagents

B-7.2.1 Standard Iodine Solution — 0.01 N (see IS 2316)

B-7.2.2 Concentrated Hydrochloric Acid — see IS 265

B-7.2.3 Standard Sodium Thiosulphate Solution — 0.01 N (see IS 2316)

B-7.2.4 Starch Indicator Solution

Triturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into 1 litre of boiling water. Boil for 3 min. Allow to cool and decant off the supernatant clear liquid.

B-7.3 Procedure

Weigh accurately about 5 g of the material, add 100 ml of water, warm slightly and filter. Wash the residue with hot water and cool the filtrate. Transfer the filtrate to a conical flask containing a measured quantity of 50 ml of standard iodine solution, then add 10 ml of concentrated hydrochloric acid and titrate the excess of iodine with standard sodium thiosulphate solution using starch indicator. Carry out the blank determination simultaneously under similar conditions.

B-7.4 Calculation

Water soluble sulphides (as BaS), percent by mass

$$= \frac{0.0847 \times (V_1 - V_2)}{M_1}$$

where

V_1 = volume, in ml, of standard sodium thiosulphate solution used in the blank titration;

V_2 = volume, in ml, of standard sodium thiosulphate solution used in the titration; and

M_1 = mass, in g, of the material taken for the test.

B-8 ESTIMATION OF TOTAL SULPHUR AS SULPHATE (SO₄)

B-8.1 Apparatus

B-8.1.1 Beaker — 400 ml capacity

B-8.1.2 Platinum Crucible — 25 ml

B-8.1.3 Volumetric Flask — 250 ml

B-8.2 Reagents

B-8.2.1 Concentrated Nitric Acid — see IS 264

B-8.2.2 Concentrated Hydrochloric Acid — see IS 265

B-8.2.3 Bromine (saturated) — see IS 2142

B-8.2.4 Sodium Carbonate — AR grade

B-8.2.5 Potassium Nitrate — AR grade

B-8.2.6 Methyl Orange

Dissolve 0.1 g of methyl orange in 60 ml of rectified spirit and dilute with water to 100 ml.

B-8.2.7 Barium Chloride Solution — 0.25 M

B-8.2.8 Dilute Hydrochloric Acid — 0.1 N

B-8.2.9 Concentrated Hydrofluoric Acid — AR

B-8.2.10 Concentrated Sulphuric Acid — see IS 266

B-8.3 Procedure

Weigh exactly 5 g of the sample and transfer the sample to a 400 ml beaker. Make it to a paste with water. Then add 15 ml conc. HNO₃, 5 ml of concentrated HCl and 0.5 ml of bromine. Boil and allow to stand the covered beaker on steam bath for 30 min. Evaporate to dryness. Add 10 drops of con. HCl, and dilute it to 200 ml with water. Heat to the boiling point. Allow the covered beaker to stand overnight. Filter through a close filter paper (Whatman 42). Wash with hot water. Discard the filtrate. Ash the precipitate in the paper in a platinum crucible. Fuse the mixture with 3 g of AR sodium carbonate (Na₂CO₃) and 0.5 g of AR potassium nitrate (KNO₃).

B-8.3.1 Leach the melted mass with water. Transfer the contents of the crucible to a 250 ml volumetric flask. Fill up to the mark with water and mix. Filter through dry paper (discard the first 10 ml) into a dry volumetric flask of 200 ml up to the mark. Transfer the contents of the 200 ml volumetric flask quantitatively to a 600 ml beaker. Add few drops of methyl orange. Acidify with 6 N HCl. Boil to remove CO₂. Neutralize and acidify with 0.1 N HCl. Heat to boil and add while stirring 5 ml of 0.25 M BaCl₂ solution. Allow for overnight settling. Filter the precipitate in Whatman 42 paper, wash with a little hot water till free from chloride. Dry and ignite the paper in platinum crucible.

B-8.3.2 To the ash add 1 ml of 40 percent HF, and one drop of concentrated H₂SO₄. Again, ignite and weigh.

B-8.4 Calculation

Total sulphur (as SO₄), percent by mass

$$= \frac{M_1 \times 51.44}{M_2}$$

where

M_1 = mass, in g, of the precipitate; and

M_2 = mass, in g, of the material taken for test.

B-8.5 Sensitivity — 1 mg to 0.01 percent SO₄

B-9 DETERMINATION OF ALKALI AND OTHER METALS

B-9.1 Outline of the Method

Alkali and other metals are determined gravimetrically as sulphates.

B-9.2 Reagents

B-9.2.1 Dilute Hydrochloric Acid — approximately 5 N

B-9.2.2 Dilute Sulphuric Acid — approximately 5 N

B-9.3 Procedure

Dissolve 5 g of the material in 100 ml of water and 20 ml of dilute hydrochloric acid. Heat to boiling, add 12 ml to 15 ml of dilute sulphuric acid and allow to stand for two hours. Filter, evaporate the filtrate to dryness ignite gently and weigh the residue.

B-9.4 Calculation

Alkali and other metals (as SO₄), percent by mass

$$= \frac{100 m}{M}$$

where

m = mass, in g, of the residue; and

M = mass, in g, of the material taken for the test.

B-10 ESTIMATION OF SODIUM OXIDE BY FLAME PHOTOMETER

B-10.1 Reagents

B-10.1.1 *Sodium Chloride* — AR grade

B-10.2 Apparatus

B-10.2.1 *Volumetric Flask* — 250 ml capacity

B-10.2.2 *Flame Photometer* — with a filter for sodium

B-10.3 Stock Solution of Sodium Chloride

Dissolve 2.545 g of AR grade sodium chloride in distilled water in 1 000 ml volumetric flask and dilute to the mark. Shake well taking care that the solution does not leak or come in contact with fingers. This solution contains 1 mg of Na per ml of the solution.

B-10.4 Preparation of Calibration Curve

To prepare calibrating solution containing 2 ppm, 4 ppm, 6 ppm, 8 ppm, 10 ppm of sodium transfer 2 ml, 4 ml, 6 ml, 8 ml, 10 ml each of stock solution in 5 different (one litre volumetric flask by means of pipette and dilute up to the mark with distilled water). Pour the calibrating solution into 1 000 ml or 500 ml plastic bottles and label them as 2 ppm, 4 ppm, 6 ppm, 8 ppm, 10 ppm of sodium respectively. The label may be covered with a transparent cellotape, so that it does not come out after some time.

Bring the sodium filter in flame photometer in position. The galvanometer spot is adjusted to left hand zero, of the scale of galvanometer when distilled water is atomized. Adjust the spot to 100 or 200 as required by atomizing highest concentration (10 ppm solution). Take the reading in quick succession of the other calibrating solution. Atomize distilled water in between the readings. It is better to repeat the series of readings to feel safe about it.

After all the readings are over, plot the graph of concentration on X axis, and galvanometer reading on Y axis. This curve is used for determining the sodium concentration of unknown solution.

B-10.5 Procedure

Take 5 g of BaCO_3 powder in a 400 ml beaker. Make it to a paste. Add drop by drop con. AR HCl, to dissolve the BaCO_3 powder. When all the carbonate

dissolved, just neutralize the solution to pH 7 with ammonia solution. Keep the solution in a water bath so that excess ammonia expels out. Now filter the solution through Whatman 42 filter paper into a 250 ml standard volumetric flask, wash well and make up the solution to 250 ml mark. Now take this solution and atomize it in the flame photometer using the sodium filter and note the galvanometer reading for the unknown solution.

From the curve we have already plotted, find out the concentration of sodium for the corresponding galvanometer reading noted in the galvanometer for the unknown solution.

B-10.6 Calculation

Sodium (as Na) parts per million

$$= \frac{\text{Concentration} \times 250}{5 \text{ g}}$$

Sodium oxide (as Na_2O), ppm = Na \times 1.35

B-11 DETERMINATION OF IRON

B-11.1 General

Three methods are prescribed for determining iron, namely, Method A, ICP-OES method as prescribed at [B-15](#) and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

B-11.2 Method A

Iron is determined colorimetrically by visual comparison.

B-11.2.1 Apparatus

B-11.2.1.1 *Nessler cylinders* — 100 ml capacity (see IS 4161)

B-11.2.2 Reagents

B-11.2.2.1 *Concentrated hydrochloric acid* — see IS 265

B-11.2.2.2 *Ammonium persulphate* — solid

B-11.2.2.3 *Butanolic potassium thiocyanate solution*

Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient n-butanol to make up to 100 ml and shake vigorously until the solution is clear.

B-11.2.2.4 *Standard iron solution*

Dissolve 0.702 g of ferrous ammonium sulphate [$\text{FeSO}_4 \cdot (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}$] in water, add 10 ml of concentrated hydrochloric acid and dilute to one

litre. One millilitre of this solution is equivalent to 0.1 mg of iron (as Fe).

B-11.2.3 Procedure

Weigh accurately about 0.25 g of the material and dissolve in 10 ml of concentrated hydrochloric acid and dilute with 10 ml of water. Add 30 mg of ammonium persulphate, warm and filter into a Nessler cylinder. Cool and add 5 ml of butanolic potassium thiocyanate solution. Add 5 ml of butanolic potassium thiocyanate to another Nessler cylinder containing 10 ml of concentrated hydrochloric acid, 30 mg of ammonium persulphate and 10 ml of water. Run in standard iron solution into the second Nessler cylinder from a burette until after thorough mixing the colours in the two Nessler cylinders are matched.

B-11.2.4 Calculation

$$\text{Iron (as Fe}_2\text{O}_3\text{), percent by mass} = \frac{0.0143 \times V}{M}$$

where

V = volume, in ml, of standard iron solution required; and

M = mass, in g, of the material taken for the test.

B-12 TAP DENSITY

B-12.1 Apparatus

B-12.1.1 Dried Measuring Cylinder — 100 ml capacity

B-12.1.2 Rubber Base Pad

B-12.1.3 Dry Funnel

B-12.2 Procedure

Take a sufficient quantity of the dried sample and slip it gently and smoothly through the funnel into the measuring cylinder, up to the 100 ml mark without knocking. Give constant tapping on the rubber base pad, slowly and smoothly, so that no sample is getting out while giving tapping. When a constant volume is obtained note down the volume and transfer the sample and weigh the sample accurately.

B-12.3 Calculation

$$\text{Tap density, g/ml} = \frac{M}{V}$$

where

M = mass, in g, of the material; and

V = volume, in ml, of the material.

B-13 RESIDUE ON SIEVE

B-13.1 Apparatus

B-13.1.1 Sieve — 63 micron IS sieve

B-13.2 Reagents

B-13.2.1 Refined Linseed Oil — see IS 75

B-13.2.2 Petroleum Hydrocarbon Solvent — 145/205

B-13.3 Procedure

Weigh accurately the appropriate quantity of the sample dried at $(110 \pm 2)^\circ\text{C}$ for 2 h. Transfer the sample to a glazed porcelain plate. Thoroughly incorporate a sufficient quantity of refined linseed oil with a palletle. Knife without any grinding action until a uniform paste is obtained. Mix the paste with 100 ml of petroleum hydrocarbon solvent 145/205 and transfer the mixture on to a 63 micron IS sieve by rinsing with further quantities of petroleum hydrocarbon solvent. Wash the residue left on the sieve with petroleum hydrocarbon solvent gently brushing with a soft camel hair brush until the washings are clear. Dry the residue to a constant mass at $(100 \pm 2)^\circ\text{C}$.

B-13.3.1 Express the result at the percentage of the mass of the material taken for the test.

B-14 REACTION EFFICIENCY

B-14.1 Reagents

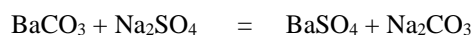
B-14.1.1 Dilute Sodium Sulphate Solution — 2 percent

B-14.1.2 Standard Hydrochloric Acid — 1 N

B-14.1.3 Methyl Orange Indicator

B-14.2 Procedure

Take 1 g of the sample and add to it dilute sodium sulphate solution in a 250 ml beaker. Boil for 45 min. Cool and filter. Titrate the filtrate against standard hydrochloric acid using methyl orange as indicator, the following reaction takes place:



$$(1 \text{ g of BaCO}_3 = 0.537 \text{ g of Na}_2\text{CO}_3)$$

$$1 \text{ ml of 1 N HCl} = 53 \text{ g of Na}_2\text{CO}_3)$$

B-14.3 Calculation

Reaction efficiency, percent by mass

$$= V \times 987$$

where

V = volume, in ml, of standard hydrochloric acid required.

B-15 DETERMINATION OF IRON BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

B-15.1 Principle

The sample solution under analysis is nebulized through a nebulizer inside a spray chamber. The aerosol formed is aspirated to argon plasma torch produced by a radio-frequency inductively coupled plasma (ICP)], where the molecules break into constituent atoms and/or molecular species and atoms are get excited. These excited atoms then return back to the lower energy state by emitting radiation of specific wavelength. These emitted radiations are characteristic of an element and are measured by the Photomultiplier tube detector and intensity of such emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

B-15.2 Recommended Wavelength, limit of quantification and important spectral interferences

Elements along with the recommended wavelengths and typical estimated limits of quantification are listed in Table 2. Actual working detection limits are dependent on the type of instrumentation, detection device and sample introduction system used and on the sample matrix. Therefore, these concentrations can vary between different instruments.

Additionally, [Table 2](#) lists the most important spectral interferences at the recommended wavelengths for analysis.

B-15.3 Reagents and Solutions

B-15.3.1. Nitric Acid (65 percent) Suprapure

B-15.3.2 Standard Stock Solution

Either prepare by dissolving proportionate amount of soluble compounds of elements (preferably spectroscopic grade), or use commercially available certified stock solution of 10 µg/ml, 100 µg/ml or 1 000 µg/ml of Lead, Iron, calcium, magnesium, manganese, arsenic, molybdenum, aluminium and mercury in 2 percent to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

B-15.3.3 Standard Solution

Pipette out 5 ml from 100 µg/ml standard stock solution into a 100 ml volumetric flask and make up volume with 2 percent nitric acid to prepare 5 µg/ml solution. From this 5 µg/ml solution, an aliquot of 1.0 ml, 3.0 ml and 5.0 ml taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1 µg/ml, 0.3 µg/ml and 0.5 µg/ml solution of respective elements under reference.

B-15.3.4 Sample preparation

Weigh about 2.5 g of the sample in a 50 ml volumetric flask and add 1.0 ml nitric acid and make up the volume with water.

NOTE — Sample should be clear before injecting to the instrument.

Table 2 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences

(Clauses [B-15.2](#) and [B-15.4](#))

Sl No.	Element	Wavelength (nm)	Approximately Achievable Limits		Interfering Elements
			Radial Viewing (µg)	Axial Viewing (µg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-

B-15.3.5 Reagent Blank Solution

Place 50 ml of nitric acid and 1 000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluoroethylene (PTFE) containers should be used. Prior to analysis, make sure that the acid matrix and concentration of the reagent blank solution is the same as in the standard and sample solutions.

B-15.4 Instrument

Set up the instrument as per the manufacturer's instructions manual for recommended operating parameters, based on the manufacturers operating manual and evaluated by internal check analysis using of standard solution of element as well as data selected carefully from [Table 2](#).

For analysis of mercury and arsenic, a gas (hydride)/vapour generating system is coupled with ICP, instead of using of nebulizer. The mercury vapour/arsine generated through the system is carried by the carrier gas (Ar) to plasma torch, and other instrumental conditions shall be the same as above.

NOTE — Sensitivity, instrumental detection limit, precision, linear dynamic range and interference effects will be investigated and established for each individual analyte line on that particular instrument.

B-15.5 Procedure**B-15.5.1 Calibration**

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions ([B-15.3.5](#)). The relationship between concentration and intensity is linear up to six orders of magnitude. Examine the spectra of the element and make necessary adjustments (if required) for the

exact peak positions and baselines to ensure proper measurements of the respective peak intensities. Flush the system with the reagent blank solution between each standard.

B-15.5.2 Before beginning the sample run, re-analyse the reference standard with the highest concentration as if it were a sample. Ensure that the concentration values do not deviate from the actual values by more than ± 5 percent (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition. Begin the sample run by flushing the system with the reagent blank solution between each sample. It is recommended to analyse a calibration check solution and the calibration blank solution every 10 samples. Analyze the sample solution and calculate the concentration in $\mu\text{g/ml}$ of the Iron in the sample solution.

NOTE — It is recommended that IS 3025(Part 2)/ISO 11885 may be referred and practiced for ensuring precise and reproducible analysis.

B-15.6 Calculation

The mass concentrations for each element are determined with the aid of the instrument software by following steps:

- a) Relate emission signals from calibration blank and calibration solutions with the signals from reference elements and establish a calibration plot; and
- b) Determine the mass concentrations of samples with the aid of the emissions and the calibration graphs and calculate the quantity in mg/kg of the constituent elemental impurities in the sample, by multiplying the value by 20 (Dilution factor).

ANNEX C

(Clause 6)

SAMPLING OF PRECIPITATED BARIUM CARBONATE, TECHNICAL

C-1 GENERAL REQUIREMENTS OF SAMPLING

C-1.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

C-1.2 Samples shall be drawn in a protected place not exposed to damp air, dust or soot.

C-1.3 The sampling instrument shall be clean and dry when used.

C-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples, from adventitious contamination.

C-1.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

C-1.6 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

C-1.7 The samples containers shall be of such a size that they are almost completely filled by the sample.

C-1.8 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

C-1.9 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

C-2 SCALE OF SAMPLING**C-2.1 Lot**

All the packages in a single consignment of the material and from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of packages from different batches of manufacture, the batches shall be marked separately and the packages in each such batch shall constitute separate lots.

C-2.2 The number of packages to be selected from the lot shall depend upon the size of the lot and shall be in accordance with [Table 3](#).

C-2.3 These packages shall be selected at random from the lot and in order to ensure the randomness of selection random number tables may be used. In case random number tables are not available, the following procedure may be adopted.

'Starting from any package in the lot count them as 1,2,3 ... up to r and so on in one order, where r is integral part of N/n (N being the lot size and n being the number of packages to be selected). Every r^{th} package thus counted shall be withdrawn to give sample for test.

Table 3 Number of Packages to be Selected(Clause [C-2.2](#))

SI No.	Lot Size	Number of Packages to be Selected
	N	n
(1)	(2)	(3)
i)	Up to 100	5
ii)	101 to 300	6
iii)	301 to 500	7
iv)	501 to 800	8
v)	801 to 1 300	9
vi)	1 301 and above	10

C-3 TEST SAMPLES AND REFEREE SAMPLES

C-3.1 Draw with an appropriate sampling instrument small portions of the material from different parts of the packages in the sample. The total quantity of material so drawn from each package shall be approximately three times the quantity required for carrying out the tests for all the requirements.

C-3.2 Mix thoroughly the portions drawn from the same package to form an individual sample representative of the package. Also mix together equal portions from the individual samples to form a composite sample representative of the whole lot.

C-3.3 Divide the individual samples and the composite sample into three sets of test samples, each set shall have individual samples representing each package and a composite sample. Transfer these test samples immediately to thoroughly dried sample containers which shall then be sealed airtight and labelled with all the particulars of sampling given under [C-1.8](#). One of these sets shall be for the purchaser, another for the supplier and the third for the referee. Referee samples shall consist of a set of individual samples and a composite sample marked for the purpose. Referee samples shall bear the seals

of the purchaser and the supplier, and shall be kept at a place agreed to between the two. It shall be used in case of dispute between the two.

C-4 NUMBER OF TESTS

C-4.1 Tests for the material insoluble in hydrochloric acid, water soluble sulphides and barium shall be conducted on each of the individual samples in the set of test samples.

C-4.2 Tests for the remaining characteristics shall be conducted on the composite samples.

C-5 CRITERIA FOR CONFORMITY

C-5.1 A lot shall be declared as conforming to the requirements of this specification, if the requirements given in [C-5.2](#) and [C-5.3](#) are satisfied.

C-5.2 The individual samples, when tested for material insoluble in hydrochloric acid, water soluble sulphides and barium shall all satisfy the requirements of the specification individually.

C-5.3 For the remaining characteristics which have been tested on the composite sample, all the test results shall satisfy the corresponding requirements of the specification.

ANNEX D

(Foreword)

COMMITTEE COMPOSITION

Inorganic Chemicals Sectional Committee, CHD 01

<i>Organization</i>	<i>Representative(s)</i>
Central Salt and Marine Chemicals Research Institute, Bhavnagar	DR KANNAN SRINIVASAN (Chairperson)
Alkali Manufacturers Association of India, Delhi	SHRI K. SRINIVASAN SHRI H. S. DAS (<i>Alternate</i>)
Bhabha Atomic Research Centre, Mumbai	DR A. V. R. REDDY DR S. N. ACHARY (<i>Alternate</i>)
Central Drugs Standard Control Organization, New Delhi	SHRI C. HARIHARAN
Consumer Voice, Delhi	SHRI M. A. U. KHAN SHRI K. C. CHAUDHARY (<i>Alternate</i>)
Consumer Education & Research Centre, Ahmedabad	DR ANINDITA MEHTA DR KARTIK ANDHARIA (<i>Alternate</i>)
Delhi Jal Board, New Delhi	SHRI ASHUTOSH KAUSHIK
Directorate General of Quality Assurance (DGQA), New Delhi	DR A. K. PATRA SHRI B. S. TOMAR (<i>Alternate</i>)
Geological Survey of India, Kolkata	SHRI PVVR SARMA
Global Adsorbents Pvt Ltd, Kolkata	SHRI SANJAY DHANUKA
Grasim Industries Ltd, Nagda	SHRI ALOK SINGH SHRI PANKAJ GUPTA (<i>Alternate</i>)
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