

भारतीय मानक मसौदा
बेरियम क्लोराइड — विशिष्टि
(IS 5288 का दूसरा पुनरीक्षण)

Draft Indian Standard
Barium Chloride — Specification
(*Second Revision of IS 5288*)

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ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01

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FOREWORD

(*formal clauses will be added later*)

This standard was first published in 1969 and subsequently revised in 1980. In the first revision, changes were made in the requirements for assay of the material, namely, technical and analytical reagent grade; for matter insoluble in water, heavy metals and iron for technical grade. Additional requirements for sodium and potassium were incorporated and a volumetric method for assay was incorporated as an alternate to the gravimetric method as the former is quicker.

In this revision, instrumental test methods for the determination of copper, iron and lead have been added as alternate test methods. In addition to this, editorial corrections have been made wherever required. Also, Amendment No.1 and Reference clause have been incorporated. Further, Packing and Marking clause has been updated.

Barium chloride is one of the most important barium chemicals, excluding those which are used for pigments. It is used for the manufacture of pigments, colour lakes, as a mordant for acid dyes; and in weighting and dyeing textile fabrics. It is used in large quantities for removal of sulphate ions from sodium chloride brine used in the electrolytic cells for the manufacture of caustic soda, pure salts, etc. It is also used in boiler compounds for softening water and in the production of other barium compounds and heat treatment salts. The other uses are in tanning of white leather and as an ingredient in rat and vermin poisons.

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.

Draft Indian Standard
BARIUM CHLORIDE — SPECIFICATION
(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for barium chloride.

2 REFERENCES

The standards given below contain provisions which, through reference in this text, constitute provision 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 edition of these standards:

| <i>IS No.</i> | <i>Title</i> |
|--|--|
| IS 264 : 2005 | Nitric acid — Specification (<i>third revision</i>) |
| IS 265 : 2021 | Hydrochloric acid — Specification (<i>fifth revision</i>) |
| IS 266 : 2024 | Sulphuric acid — Specification (<i>fourth revision</i>) |
| IS 321 : 1964 | Specification for absolute alcohol (<i>first revision</i>) |
| IS 1070 : 2023 | Reagent grade water — Specification (<i>fourth revision</i>) |
| IS 3025 (Part 2) : 2019/ISO 11885 : 2007 | Methods of sampling and test (physical and chemical) for water and wastewater Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP - OES) (<i>first 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 4161 : 2023 | Nessler cylinder — Specification (<i>first revision</i>) |

3 GRADES

There shall be two grades of the material, namely,

- a) Technical (TECH); and
- b) Analytical Reagent (AR).

4 REQUIREMENTS**4.1 Description**

The material shall be in the form of colourless crystals, free from extraneous impurities.

4.2 The material shall also comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col **5** of the table.

5 PACKING AND MARKING**5.1 Packing**

5.1.1 Technical grade of the material shall be packed in clean jute bags lined with polyethylene.

5.1.2 Analytical reagent grade of the material shall be packed in glass bottles.

5.2 Marking

Each container shall bear legibly and indelibly the following information:

- Name and grade of the material;
- Name of the manufacturer and/or his recognized trade-mark, if any;
- Net mass of the contents;
- Date of manufacture; and
- Batch number.

5.2.1 In case of AR grade of the material, complete chemical analysis in respect of the characteristics specified in Table 1 shall also appear on the label.

5.2.2 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.

Table 1 Requirements for Barium Chloride

(Clauses 4.2 and 5.2.1)

| Sl. No. | Characteristic | Requirement | | Method of Test, Ref to CI No. in Annex A |
|---------|---|---------------|---------------|--|
| | | Tech Grade | AR Grade | |
| (1) | (2) | (3) | (4) | (5) |
| i) | Assay (as BaCl ₂ .2H ₂ O), percent by mass | 99.0 to 102.0 | 99.0 to 101.0 | A-2 |
| ii) | Matter insoluble in water, percent by mass, <i>Max</i> | 0.1 | 0.005 | A-3 |
| iii) | Oxidizing substances (as NO ₃), percent by mass, <i>Max</i> | - | 0.002 | A-4 |
| iv) | Heavy metals (as Pb), mg/kg, <i>Max</i> | 100 | 3 | A-5 |
| v) | Iron (as Fe), percent by mass, <i>Max</i> | 0.002 | 0.000 1 | A-6 |
| vi) | Substances not precipitated by sulphuric acids, percent by mass, <i>Max</i> | 0.2 | 0.02 | A-7 |
| vii) | Calcium and strontium salts, percent by mass, <i>Max</i> | - | 0.10 | A-8 |
| viii) | pH (of aqueous solution) | 5.0 to 8.0 | 5.0 to 8.0 | A-9 |
| ix) | Water soluble sulphides (as S), mg/kg, <i>Max</i> | 20 | - | A-10 |
| x) | Sodium (as Na), percent by mass, <i>Max</i> | - | 0.02 | * |
| xi) | Potassium (as K), percent by mass, <i>Max</i> | - | 0.02 | * |
| xii) | #Sulphur compounds (as S) | To pass test | To pass test | A-11 |
| xiii) | #Copper (as Cu), percent by mass, <i>Max</i> | 0.002 | - | A-12 |

* Determine sodium and potassium by flame photometer at 589 m μ and 766.5 m μ respectively according to directions of manufacturer of the apparatus.

These are additional requirements for technical grade of the material, when it is to be used for producing heat treatment salts.

6 SAMPLING

The method of drawing representative samples of the material, number of tests to be performed and the method of determining the conformity of the material to the requirements of this specification shall be as prescribed in Annex B.

ANNEX A

(Clause 4.2)

METHODS OF TEST FOR BARIUM CHLORIDE

A-1 QUALITY OF REAGENTS

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

NOTE – ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF BARIUM CHLORIDE

A-2.1 Barium chloride can be determined gravimetrically by weighing as barium chromate, or the precipitated barium chromate can be dissolved in hydrochloric acid and volumetric estimation can be done as a quick alternative method. The gravimetric method shall be followed as a referee method.

A-2.2 Gravimetric

A-2.2.1 Reagents

A-2.2.1.1 Dilute acetic acid — 5 N.

A-2.2.1.2 Ammonium chloride — solid.

A-2.2.1.3 Potassium chromate solution — 5 percent (*m/v*).

A-2.2.2 Procedure

Weigh accurately 5 g of the material, dissolve it in 100 ml of water, filter, wash and make up the solution to 500 ml with water in a volumetric flask. Take 25 ml to aliquot of the solution, add 2 ml of dilute acetic acid, 5 g of ammonium chloride and heat to boiling. Add 20 ml of potassium chromate solution slowly with constant stirring and allow to stand for 2 h. Filter the precipitate through a tared sintered glass crucible No. G4, and wash with cold water until the washings are free from chlorides. Dry the crucible at $(130 \pm 5)^\circ\text{C}$ to constant mass, cool in a desiccator and weigh.

A-2.2.3 Calculation

$$\text{Barium chloride (as BaCl}_2 \cdot 2\text{H}_2\text{O), percent by mass} = \frac{M_1 \times 96.42}{M}$$

where

M_1 = mass in g of the dried residue; and

M = mass in g of the material present in the aliquot.

A-2.3 Volumetric

A-2.3.1 Reagents

A-2.3.1 Hydrochloric acid — 5 N.

A-2.3.1.2 Potassium iodide crystals

A-2.3.1.3 Standard sodium thiosulphate solution — freshly standardized.

A-2.3.1.4 Starch solution

Triturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of water in a mortar. Pour the resulting paste into 1 litre of boiling water, boil for 30 min, allow the solution to cool, and decant off the clear liquid.

A-2.3.2 Procedure

Dissolve the washed precipitate of barium chromate (A-2.2.2) with 25 ml of dilute hydrochloric acid and transfer into a 250 ml glass stoppered conical flask. Add 50 ml of water and 4 g of potassium iodide. Keep the flask in dark for 10 min. Titrate the liberated iodine with standard thiosulphate solution, using about 5 ml of starch solution towards the end, until the blue colour produced just disappears.

A-2.3.3 Calculation

$$\text{Barium chloride (as BaCl}_2 \cdot 2\text{H}_2\text{O), percent by mass} = \frac{2 \times N \times V \times 81.43}{M}$$

where

N = normality of standard thiosulphate solution;

V = volume in ml of standard thiosulphate solution required in the titration; and

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

A-3 DETERMINATION OF MATTER INSOLUBLE IN WATER**A-3.1 Procedure**

Weigh accurately 20 g of the material and dissolve in 200 ml of water, heat to boiling and then digest in a covered beaker on the steam-bath for 1 h. Filter through a sintered glass crucible or Gooch crucible, wash thoroughly, and dry at (105 ± 5) °C till constant mass is obtained.

A-3.2 Calculation

$$\text{Matter insoluble in water, percent by mass} = \frac{100 \times M_1}{M}$$

where,

M_1 = mass in g of the dried residue, and

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

A-4 DETERMINATION OF OXIDIZING SUBSTANCES**A-4.1 Reagents**

A-4.1.1 Concentrated Sulphuric Acid — see IS 266.

A-4.1.2 Diphenylamine Solution

Dissolve 10 mg of diphenylamine in 100 ml of concentrated sulphuric acid. In a separate beaker, dissolve 2 g of the ammonium chloride in 200 ml of water. Cool both the solutions in ice and cautiously add the acid solution to the water taking care to keep the resulting solution cool. The final solution shall be water white.

A-4.1.3 Standard Nitrate Solution

Dissolve 0.163 g of potassium nitrate (KNO_3) in water and dilute to exactly 100 ml. Dilute 10 ml of this solution to 1 000 ml in volumetric flask. One milliliter of this solution is equivalent to 0.01 mg of nitrate (as NO_3).

A-4.1.4 Sodium Carbonate — solid.

A-4.2 Procedure

Weigh accurately 0.1 g of the material in a beaker. Place it in an ice-bath and add 20 ml of concentrated sulphuric acid which has been previously cooled to ice-bath temperature. Allow the mixture to warm to room temperature and swirl it at intervals to effect dissolution with gentle evolution of hydrogen chloride. When dissolution is complete, add 3 ml of diphenylamine solution and digest on a steam-bath for 90 min. Prepare a control by evaporating to dryness 0.5 ml of standard nitrate solution and 0.01 g of sodium carbonate. Treat the residue in the same manner and compare the colour produced in the two tests.

A-4.2.1 The material shall be considered to have not exceeded the limit prescribed in Table 1 if the colour produced by the material is not greater than that produced in the control test.

A-5 DETERMINATION OF HEAVY METALS (as Pb)

A-5.1 General

Three methods are prescribed for determining heavy metals (as Pb), namely, Method A, ICP-OES method as prescribed at **A-13** and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

A-5.2 Method A

A-5.2.1 Apparatus

A-5.2.2.1 *Nessler cylinders* — 50 ml capacity (see IS 4161).

A-5.2.2 Reagents

A-5.2.2.1 Standard lead solution

Dissolve 1.60 g of lead nitrate in water and make up the volume to exactly 1 000 ml. Transfer exactly 10 ml of the solution to a volumetric flask and dilute it again with water to 1 000 ml mark. One milliliter of this solution contains 0.01 mg of lead (as Pb). The dilute solution shall be freshly prepared.

A-5.2.2.2 *Dilute acetic acid* — approximately 1 N.

A-5.2.2.3 *Hydrogen sulphide solution* — saturated.

A-5.2.3 Procedure

Weigh 10 g of the material and dissolve in 20 ml of water. Transfer the solution to a Nessler cylinder, add 1 ml of dilute acetic acid 10 ml of hydrogen sulphide solution. In the second Nessler cylinder, carry out a control test using 3 ml of standard lead solution and the same quantities of other reagents. Dilute the contents of each cylinder to 50 ml and shake well. Compare the colour produced in two cylinders.

A-5.2.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

A-6 DETERMINATION OF IRON

A-6.1 General

Three methods are prescribed for determining iron, namely, Method A, ICP-OES method as prescribed at **A-13** and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

A-6.2 Method A

A-6.2.1 Apparatus

A-6.2.1.1 *Nessler cylinders* — 50 ml capacity (see IS 4161).

A-6.2.2 Reagents

A-6.2.2.1 *Concentrated hydrochloric acid* — see IS 265.

A-6.2.2.2 *Concentrated nitric acid* — see IS 264.

A-6.2.2.3 *Ammonium persulphate* — solid.

A-6.2.2.4 *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.

A-6.2.2.5 *Standard iron solution*

Dissolve 0.702 g of ferrous ammonium sulphate [$\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$] in 10 ml of dilute sulphuric acid (10 percent v/v) and dilute with water to make up the volume to exactly 1 000 ml. Pipette out 10 ml of this solution and dilute with water to make up the volume to exactly 100 ml. One milliliter of this solution is equivalent to 0.01 mg of iron (as Fe).

A-6.2.3 *Procedure*

Weigh 0.1 g of the material and dissolve in 10 ml of water. Add 2 ml of hydrochloric acid and 5 drops of nitric acid and boil. Cool the solution, and 15 ml of butanolic potassium thiocyanate. Make up the solution to 50 ml. Shake vigorously for 30 s and allow the butanolic layer to separate. Carry out a control test in the second Nessler cylinder using 0.3 ml of standard iron solution in place of the material for technical grade of the material, and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour produced in the two cylinders after 2 min. For AR grade, use 1 g of the material and 0.1 ml of standard iron solution.

A-6.2.3.1 The limits prescribed in Table 1 shall be taken as not having been exceeded if the colour produced in the test with the material is not deeper than that produced in the control test.

A-7 DETERMINATION OF SUBSTANCES NOT PRECIPITATED BY SULPHURIC ACID

A-7.1 Reagents

A-7.1.1 *Concentrated Hydrochloric Acid* — see IS 265.

A-7.1.2 *Dilute Sulphuric Acid* — 1 N approximately.

A-7.2 Procedure

Weigh accurately about 5 g of the material and dissolve in 150 ml of water. Add 1 ml of concentrated hydrochloric acid, heat in 150 ml of water. Add 1 ml of concentrated hydrochloric acid, heat to boiling, and add 50 ml of dilute sulphuric acid. Cool the solution, dilute to 250 ml and allow to stand overnight. Decant through a filter paper, collecting the filtrate in a beaker. Take 100 ml of the filtrate and evaporate to dryness in a tared porcelain dish. Ignite gently to volatilize the excess acids and finally ignite at $(800 \pm 25)^\circ\text{C}$ for 15 min. Cool and weigh till constant mass is obtained.

A-7.3 Calculation

$$\text{Substances not precipitated by sulphuric acid, percent by mass} = 250 \times \frac{M_1}{M}$$

where,

M_1 = mass in g of the ignited residue; and

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

A-8 DETERMINATION OF CALCIUM AND STRONTIUM SALTS

A-8.1 Reagents

A-8.1.1 *Absolute Alcohol* — see IS 321

A-8.1.2 *Concentrated Sulphuric Acid* — see IS 266

A-8.2 Procedure

Shake about 2.5 g of the finely powered material, accurately weighed, with 50 ml of absolute alcohol for 5 min. Filter through a Whatman filter paper No. 40 in a tared silica or porcelain basin. Wash the insoluble matter twice with absolute alcohol collecting the filtrate in the same basin and evaporate it to dryness on a stem-bath. Moisten the residue in the basin and evaporate it to dryness on a stem-bath. Moisten the residue in the basin with 3 to 4 drops of concentrate sulphuric acid, ignite gently, cool in a desiccator and weigh till constant mass is obtained.

A-8.3 Calculation

$$\text{Calcium and strontium salts (as sulphates), percent by mass} = 100 \times \frac{M_1}{M}$$

where,

M_1 = mass in g of the ignited residue; and

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

A-9 DETERMINATION OF pH**A-9.1 Procedure**

Dissolve 5 g of the material in 100 ml of freshly boiled and cooled water. Determine pH of the solution by means of a suitable pH meter using glass calomel electrode.

A-10 DETERMINATION OF WATER-SOLUBLE SULPHIDES**A-10.1 Reagents**

A-10.1.1 *Standard Iodine Solution* — 0.01 N.

A-10.1.2 *Dilute Hydrochloric Acid* — 1 N approximately.

A-10.1.3 *Starch Indicator Solution*

Triturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of water in a mortar. Pour the resulting paste into 1 litre of boiling water and boil for 3 min, allow the solution to cool and decant off the clear liquid.

A-10.2 Procedure

Measure 0.2 ml of standard iodine solution into a conical flask containing 100 ml of water acidified with 2 ml of dilute hydrochloric acid. Add a few drops of starch indicator to turn it distinctly blue. Add 2 g of the barium chloride into the flask and shake to dissolve completely.

A-10.2.1 The limit prescribed in Table 1 shall be considered to have not exceeded if the blue colour does not entirely disappear.

A-11 TEST FOR SULPHUR COMPOUNDS**A-11.1 Reagents**

A-11.1.1 *Bromine Water* — saturated.

A-11.1.2 *Dilute Hydrochloric Acid* — 20 percent (v/v).

A-11.2 Procedure

Weigh accurately 5 g of the material and dissolve in 100 ml of water. Add 1 ml of dilute hydrochloric acid and 10 ml of bromine water and boil to expel liberated bromine. Continue boiling for 15 min and allow to stand below the boiling point for 30 min. The material shall be considered to have passed the test if no turbidity is produced.

A-12 DETERMINATION OF COPPER**A-12.1 General**

Three methods are prescribed for determining copper, namely, Method A, ICP-OES method as prescribed in IS 3025 (Part 2) and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

A-12.2 Method A**A-12.2.1 Apparatus**

A-12.2.1.1 Nessler cylinders — 50 ml capacity (see IS 4161)

A-12.2.2 Reagents

A-12.2.2.1 Dilute sulphuric acid — 1 : 3 (v/v).

A-12.2.2.2 Concentrated hydrochloric acid — see IS 265

A-12.2.2.3 Concentrated nitric acid — see IS 264

A-12.2.2.4 Citric acid — solid

A-12.2.2.5 Dilute ammonium hydroxide — approximately 5 N

A-12.2.2.6 Sodium diethyldithiocarbamate solution

Dissolve 1 g of diethyldithiocarbamate in 1 000 ml of water. Filter if necessary. Preserve in amber coloured glass bottle protected from light. The solution should not be used after two weeks.

A-12.2.2.7 Standard copper solution

Dissolve 0.196 g of copper sulphate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in water and make up to 1 000 ml in a volumetric flask. Pipette out 10 ml of this solution and dilute to exactly 100 ml. One milliliter of this solution contains 0.005 mg of copper (Cu). The dilute solution shall be freshly prepared.

A-12.2.3 Procedure

Weigh accurately 1 g of the material and dissolve in about 50 ml of water. Add dilute sulphuric acid to precipitate barium. Add slight excess of dilute sulphuric acid. Filter and wash the precipitate. Add to the filtrate 10 drops of concentrated hydrochloric acid and 5 drops of concentrated nitric acid and boil. Allow to cool and transfer to a Nessler cylinder. Add 1 g of citric acid and adjust the pH to 9 by addition of dilute ammonium hydroxide. Add 10 ml of sodium diethyldithiocarbamate solution and make up the volume to 50 ml mark. Carry out a control test in the other Nessler cylinder using 4 ml of standard copper solution proceeding exactly as for the material under test. Mix the contents in the two cylinders well and compare the colour produced.

A-12.2.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour produced with the material is not greater than that in the control test.

A-13 DETERMINATION OF LEAD AND IRON BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD**A-13.1 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.

A-13.2 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.

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

| SI 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 | - | - | - |
| ii) | Pb | 220.353 | 14 | 5 | Al, Co, Fe, Ti |
| | | 283.305 | (70) | | Cr, Fe |
| | | | | (20) | |

A-13.3 Reagents and Solutions**A-13.3.1. Nitric acid (65 percent) Suprapure****A-13.3.2 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, 100 or 1 000 $\mu\text{g}/\text{ml}$ of Lead, Iron, calcium, magnesium,

manganese, arsenic, molybdenum, aluminium and mercury in 2 to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

A-13.3.3 *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, 3.0 and 5.0 ml taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1, 0.3 and 0.5 µg/ml solution of respective elements under reference.

A- 13.3.4 **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.

A-13.3.5 *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, polytetrafluorethylene (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.

A-13.4 **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.

A-13.5 **Procedure**

A-13.5.1 *Calibration*

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (A-13.3.5). 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.

A-13.5.2 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 µg/ml of the lead (and/or Iron, calcium) 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.

A-13.6 **Calculation**

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

- i) Relate emission signals from calibration blank and calibration solutions with the signals from reference elements and establish a calibration plot.
- ii) 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 B

(Clause 6)

SAMPLING OF BARIUM CHLORIDE

B-1 GENERAL REQUIREMENTS OF SAMPLING

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

B-1.2 Samples shall not be taken in a place exposed to weather.

B-1.3 The sampling instrument shall be clean and dry.

B-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.

B-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.

B-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.

B-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, month and year of manufacture, and other important particulars of the consignment.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers in a single consignment of the material, of the same grade, drawn from a single batch of manufacturer shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately batches of manufacture, the batches shall be marked separately and the group of containers in each batch shall constitute separate lots. In the case of a consignment drawn from a continuous process, 1 000 containers (or 100 tonnes) of the material shall constitute a lot.

B-2.2 The number of containers to be chosen from a lot shall depend on the size of the lot and shall be as given in Table 3.

B-3 PREPARATION OF TEST SAMPLES

B-3.1 Draw with an appropriate sampling implement about 80 g of barium chloride from parts different parts of each of the selected containers.

Mix thoroughly all portions of the material from the same container to form individual test samples.

Table 3 Number of Containers to be Selected for Sampling*(Clause B-2.2)*

| Lot Size | Number of Containers to be Selected |
|---------------|-------------------------------------|
| N (1) | n (2) |
| Up to 25 | 3 |
| 26 to 50 | 4 |
| 51 to 100 | 5 |
| 101 to 200 | 6 |
| 201 and above | 8 |

B-3.2 Equal quantities from each of the individual test samples shall be mixed together to form a composite sample weighing approximately 150 g.

B-3.3 Each of the individual test samples and the composite sample shall be divided into three equal parts, forming three sets of test samples one for the purchaser, one for the supplier and the third for the referee. The three sets of individual samples and composite sample shall be transferred to thoroughly dried bottles which shall then be sealed air-tight with glass stoppers. They shall be labelled with all relevant particulars of sampling.

B-4 NUMBER OF TESTS

B-4.1 Tests for the determination of barium chloride shall be conducted on each of the individual test samples.

B-4.2 Tests for the remaining characteristics given in 4 shall be conducted on the composite test sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples

From the test results for barium chloride content the mean (\bar{X}) and range (R) shall be calculated (range being defined as the difference between the maximum and minimum of the test results).

B-5.1.1 The lot shall be declared as conforming to the requirement of barium chloride if the value of the expression ($\bar{X} \pm 0.6 R$) is between 99.0 to 102.0 for technical grade and between 99.0 to 101.0 for AR grade.

B-5.2 For Composite Sample

For declaring the conforming of the lot to the requirements of all other characteristics tested on composite sample, the test result for each of the characteristics shall satisfy the relevant requirement specified in Table 1.