

भारतीय मानक मसौदा  
पोटेशियम ब्रोमाइड — विशिष्टि  
(IS 2797 का चौथा पुनरीक्षण)

*Draft Indian Standard*  
**Potassium Bromide — Specification**  
(*Fourth Revision of IS 2797*)

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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 originally published in 1964 and subsequently revised in 1982, 1994 and 1998. In the third revision, titrimetric method for determination of chlorides, colorimetric method of heavy metals & iron and turbidimetric method for determination of sulphate were incorporated as an alternate method.

In this revision, instrumental test methods for the determination of chlorides, sulphates, iron, lead and arsenic have been added as alternate test methods. In addition to this, editorial corrections have been made wherever required. Also, Reference clause and Packing & Marking clause have been updated.

Potassium bromide is used in medicine, preparation of other bromides and special soaps, in photography, and as a reagent in laboratories. In photography, this material is used for processing photographic films and in the preparation of photographic emulsions.

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*  
**POTASSIUM BROMIDE — SPECIFICATION**  
(*Fourth Revision*)

**1 SCOPE**

This standard prescribes requirements and methods of sampling and test for potassium bromide.

**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 editions of these standards.

**3 GRADES**

The material shall be of two grades as given below:

*Grade 1* — Analytical reagent grade, and

*Grade 2* — Pure grade suitable for the chemical industry.

**4 REQUIREMENTS****4.1 Description**

The material shall be in the form of colourless, transparent or opaque crystals, or a white granular powder, free from foreign matter and visible impurities.

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

**5 PACKING AND MARKING****5.1 Packing**

Unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in clean galvanized iron drums lined with polyethylene or in glass bottles.

**5.2 Marking**

The containers shall be marked with the following details:

- a) Name and grade of the material;
- b) Indication of the source of manufacture;
- c) Mass of the material in the container;
- d) Batch number; and
- e) Date of manufacture.

**5.2.1** The containers of Grade 1 material shall also be labelled with the analytical data for the characteristics given in col 3 of Table 1.

**Table 1 Requirements for Potassium Bromide**

(Clause 4.2, 5.2.1, B-4.1.3.1, B-5.2.1, B-7.1.3.1, B-8.1.3.1, B-9.1.3.1, B-11.2.3.1, B-11.3.3.1 and C-5.1.1)

SI No.	Characteristic	Requirement for		Method of Test (Ref to Cl No. of Annex B)
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Potassium bromide, percent by mass of dried material, <i>Min</i>	99.0	98.5	<b>B-2</b>
ii)	pH of aqueous solution	6.0 to 7.5	6.0 to 7.5	<b>B-3</b>
iii)	Chlorides (as Cl), percent by mass, <i>Max</i>	0.2	0.45	<b>B-4 or B-17</b>
iv)	Bromates (as BrO <sub>3</sub> ), percent by mass, <i>Max</i>	0.001	0.001	<b>B-5</b>
v)	Iodides	To pass test		<b>B-6</b>
vi)	Heavy metals (as Pb), percent by mass, <i>Max</i>	0.001	0.001	<b>B-7 or B-16</b>
vii)	Iron (as Fe), percent by mass, <i>Max</i>	0.001	0.008	<b>B-8 or B-16</b>
viii)	Sulphates (as SO <sub>4</sub> ), percent by mass, <i>Max</i>	0.01	0.06	<b>B-9 or B-17</b>
ix)	Moisture, percent by mass, <i>Max</i>	0.5	1.0	<b>B-10</b>
x)	Alkaline earth metals (as Mg). percent by mass, <i>Max</i>	0.005	0.01	<b>B-11</b>
xi)	Sodium (as Na), percent by mass, <i>Max</i>	0.05	—	<b>B-12</b>
xii)	Arsenic (as As <sub>2</sub> O <sub>3</sub> ), ppm, <i>Max</i>	5	10	<b>B-13 or B-16</b>
xiii)	Barium	—	To pass test	<b>B-14</b>
xiv)	Matter insoluble in water, percent by mass, <i>Max</i>	0.005	—	<b>B-15</b>

**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.

**6 SAMPLING**

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

## ANNEX A

*(Clause 2)***(LIST OF REFERRED STANDARDS)**

<i>IS No.</i>	<i>Title</i>
IS 264 : 2005	Nitric acid — Specification ( <i>third revision</i> )
IS 323 : 2009	Rectified spirit for industrial use — Specification ( <i>second revision</i> )
IS 1070 : 2023	Reagent grade water — Specification ( <i>fourth revision</i> )
IS 2088 : 2023	Methods for determination of arsenic ( <i>second revision</i> )
IS 4161 : 2023	Nessler cylinder — Specification ( <i>first revision</i> )
IS 3025(Part 2) : 2019/ISO 11885	Methods of sampling and test (physical and chemical) for water and wastewater: Part 2 determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP - OES) ( <i>first revision</i> )
IS 3025 (Part 65) : 2022	Methods of sampling and test physical and chemical for water and wastewater part 65 application of inductively coupled plasma mass spectrometry ( ICP-MS ) — Determination of selected elements including uranium isotopes ( <i>first revision</i> )
IS 4905: 2015/ ISO 24153: 2009	Random sampling and randomization procedures ( <i>first revision</i> )

## ANNEX B

(Clause 4.2, and Table 1)

## METHODS OF TEST FOR POTASSIUM BROMIDE

## B-1 QUALITY OF REAGENTS

**B-1.1** Unless specified otherwise, 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 POTASSIUM BROMIDE

## B-2.1 Reagents

**B-2.1.1** *Standard Silver Nitrate Solution* — 0.1 N.

**B-2.1.2** *Concentrated Nitric Acid* — *see* IS 264.

**B-2.1.3** *Nitrobenzene*

**B-2.1.4** *Ferric Alum Indicator Solution Saturated Solution.*

**B-2.1.5** *Standard Ammonium Thiocyanate Solution* — 0.1 N.

## B-2.2 Procedure

Dry the material to constant mass at  $(105 \pm 2)$  °C. Weigh accurately about 0.4 g of the dried material and dissolve in 50 ml of water. Add with a pipette 50 ml of standard silver nitrate solution, 2 ml of concentrated nitric acid, 1.5 ml of nitrobenzene and finally 2 ml of ferric alum indicator solution. Titrate the solution with standard ammonium thiocyanate solution to the first persistent colour change.

## B-2.3 Calculation

$$\text{Potassium bromide (as KBr), percent by mass, of dried material} = \frac{1.19 (50 - V)}{M}$$

where

$V$  = volume in ml of standard ammonium thiocyanate solution used in the titration, and

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

NOTE — Presence of chlorides increases the results for potassium bromide and the results shall therefore be corrected. One gram of chloride (as Cl) is equivalent to 3.35 g of potassium bromide.

## B-3 DETERMINATION OF pH OF AQUEOUS SOLUTION

## B-3.1 Procedure

Dissolve 5 g of the material in 100 ml of freshly boiled and cooled water (the water shall be protected with a soda lime tube while cooling). Determine the pH of the solution using a suitable pH meter.

## B-4 TEST FOR CHLORIDES

Two methods are prescribed for determination of chloride. Method A shall be the referee method and Method B the alternative method.

## B-4.1 Method A

## B-4.1.1 Apparatus

**B-4.1.1.1** *Nessler cylinder* — of 50 ml capacity (*see* IS 4161).

## B-4.1.2 Reagents

**B-4.1.2.1** *Dilute nitric acid* — 1:2 (v/v).

**B-4.1.2.2 Hydrogen peroxide**

Dilute 1 volume of 30 percent hydrogen peroxide with 1 volume of water.

**B-4.1.2.3 Concentrated nitric** — same as in **B-2.1.2**.

**B-4.1.2.4 Silver nitrate solution** — 10 percent acid.

**B-4.1.2.5 Standard chloride solution**

Dissolve 0.164 9 g of sodium chloride in water and make up the volume to 1 000 ml. Dilute 100 ml of the solution to 1 000 ml. One millilitre of the solution contains 0.01 mg of chloride (as Cl).

**B-4.1.3 Procedure**

Dissolve 0.5 g of the material in 15 ml of dilute nitric acid in a small conical flask. Add 6 ml of hydrogen peroxide and digest on a steam-bath until the solution is colourless. Wash down the sides of the flask with water, digest again for 15 min, cool and dilute to 250 ml with water. Pipette out 10 ml of the solution into a Nessler cylinder, dilute to about 25 ml with water, add 1 ml of concentrated nitric acid and 0.5 ml of silver nitrate solution. Dilute to 50 ml and mix well. Carry out a control test in another Nessler cylinder using 4 ml of standard chloride solution in the case of Grade 1 and 9.2 ml of standard chloride solution in the case of Grade 2 of the material and the same quantities of the other reagents.

**B-4.1.3.1** The material shall be taken as not having exceeded the limit given in Table 1 if the turbidity produced with the material is not greater than that produced in the control test.

**B-4.2 Method B****B-4.2.1 Outline of the Method**

This method specifies a titrimetric method for the determination of chlorides in potassium bromide.

**B-4.2.2 Principle**

The sample is treated with dilute nitric acid and hydrogen peroxide to eliminate the bromide to prevent its interference and the chloride is titrimetrically determined by back titration using ammonium thiocyanate solution after precipitating out chloride by adding excess silver nitrate solution.

**B-4.2.3 Apparatus****B-4.2.3.1 Standard laboratory apparatus****B-4.2.4 Reagent****B-4.2.4.1 Distilled water****B-4.2.4.2 Dilute nitric acid**

Approximately 8 mol/l. Pour 50 ml of concentrated nitric acid (sp. gr 1.42) into 50 ml of distilled water and mix.

**B-4.2.4.3 Hydrogen peroxide**

Approximately 15 percent (*m/v*). Dilute 50 ml of 30 percent hydrogen peroxide with 50 ml of distilled water.

**B-4.2.4.4 Concentrated nitric acid** — specific gravity of 1.42.

**B-4.2.4.5 Standard silver nitrate solution** — 0.1 mol/l.

Dry silver nitrate ( $\text{AgNO}_3$ ) at 105 °C for one hour. Dissolve 8.494 g in distilled water and make up to 500 ml in a 500 ml volumetric flask and mix. Store in an amber coloured bottle away from light.

**B-4.2.4.6 Ferric ammonium sulphate solution** [ $\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ ]

Saturated solution in distilled water (approximately 40 percent solution and acidified with a few drops of nitric acid).

**B-4.2.4.7 Standard ammonium thiocyanate solution**

0.1 mol/l. Dissolve 7.513 g of the salt ( $\text{NH}_4\text{CNS}$ ) in distilled water in a beaker, transfer quantitatively into a 1 litre volumetric flask, make up to the mark and mix.

#### **B-4.2.5 Procedure**

Weigh about 5 g of potassium bromide (correct to 1 mg) and transfer into a 100 ml conical flask. Dissolve in 30 ml of dilute nitric acid (**B-4.2.4.2**). Add 15 ml of dilute hydrogen peroxide solution (**B-4.2.4.3**) and digest on a steam-bath until the solution becomes colourless. Wash down the sides of the flask with distilled water and digest again for 15 min and cool. Now add 1 ml of ferric ammonium sulphate solution (**B-4.2.4.6**) and then add dropwise (with continuous agitation) concentrated nitric acid (**B-4.2.4.4**) until the reddish brown precipitate formed dissolves, cool and add 10.0 ml of standard silver nitrate solution (**B-4.2.4.5**) with continuous agitation. Let this volume be  $V_1$  ml.

NOTE — The volume of silver nitrate added should be more than the volume required to precipitate chloride completely.

Filter the contents of the conical flask into another 100 ml conical flask using a Whatman No. 40 filter paper. Wash the precipitate on the filter paper with two 5 ml portions of distilled water. Titrate the filter with standard ammonium thiocyanate solution (**B-4.2.4.7**) to the first appearance of reddish brown colour lasting for a minimum thiocyanate solution consumed (let this be  $V_2$  ml).

#### **B-4.2.6 Calculation and Expression of results**

The chloride content as Cl is calculated from the formula given below:

$$\text{Chloride content as Cl (percent by mass)} = \frac{(V_1 - V_2) \times 0.3547}{M_0}$$

where

$V_1$  = volume in ml of 0.1 mol/l silver nitrate added,

$V_2$  = volume in ml of 0.1 mol/l ammonium thiocyanate consumed for titration, and

$M_0$  = mass in grams of potassium bromide sample used.

NOTE — 1 ml of 0.1 mol/l  $\text{NH}_4\text{CNS}$  = 1 ml of 0.1 mol/l

$\text{AgNO}_3 = 0.003547 \text{ g Cl}$ .

#### **B-4.3 Alternative Method**

Chlorides may alternatively be determined by instrumental test method as prescribed at **B-17**.

### **B-5 TEST FOR BROMATES**

#### **B-5.1 Reagents**

**B-5.1.1 Potassium Iodide Solution** — 10 percent.

**B-5.1.2 Starch Solution**

**B-5.1.3 Dilute Sulphuric Acid** — 1 : 15 (v/v).

#### **B-5.2 Procedure**

Dissolve 1 g of the material in 10 ml of water. Add 2 drops of potassium iodide solution, 1 ml of starch solution and 5 drops of dilute sulphuric acid. Mix thoroughly and allow to stand for 10 min.

**B-5.2.1** The material shall be taken as not having exceeded the limit prescribed in Table 1 if no blue colour is produced.

### **B-6 TEST FOR IODIDES**

#### **B-6.1 Reagents**

**B-6.1.1 Carbon Tetrachloride or Chloroform**

**B-6.1.2 Ferric Chloride Solution** — 10 percent.

**B-6.1.3 Dilute Sulphuric Acid** — 1 : 9 (v/v).

### **B-6.2 Procedure**

Dissolve 5 g of the material in 20 ml of water. Add 1 ml of chloroform or carbon tetrachloride, 3 drops of ferric chloride solution and 5 drops of dilute sulphuric acid. Shake the mixture vigorously.

**B-6.2.1** The material shall be taken to have passed the test if no violet colour is produced in the chloroform or carbon tetrachloride layer.

## **B-7 TEST FOR HEAVY METALS**

Two methods are prescribed. Method A shall be referee method and Method B the alternative method.

### **B-7.1 Method A**

#### **B-7.1.1 Apparatus**

**B-7.1.1.1 Nessler cylinders** — 50 ml capacity (see IS 4161).

#### **B-7.1.2 Reagents**

##### **B-7.1.2.1 *p*-Nitrophenol indicator solution**

Dissolve 0.2 g of *p*-Nitrophenol in hot water and dilute to 100 ml.

**B-7.1.2.2 Dilute ammonium hydroxide** — 1 : 9 (v/v).

**B-7.1.2.3 Dilute hydrochloric acid** — 1 : 99 (v/v).

**B-7.1.2.4 Hydrogen sulphide solution** — saturated and freshly prepared.

##### **B-7.1.2.5 Standard lead solution**

Dissolve 1.60 g of lead nitrate in water and make up the solution to 1 000 ml with water. Pipette out 10 ml of the solution and dilute again to 1 000 ml with water. One millilitre of this solution contains 0.01 mg of lead (as Pb).

#### **B-7.1.3 Procedure**

Dissolve 2 g of the material in 25 ml of water in a Nessler cylinder and add 1 drop of *p*-Nitrophenol indicator solution. Add dropwise dilute ammonium hydroxide until the solution turns yellow. Add dilute hydrochloric acid dropwise until the solution becomes colourless and then add 0.5 ml of the acid in excess. Add 5 ml of hydrogen sulphide solution, dilute to the mark and mix well. Carry out a control test in a similar manner in another Nessler cylinder using 1 ml of standard lead solution.

**B-7.1.3.1** The relevant 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.

### **B-7.2 Method B**

#### **B-7.2.1 Outline of the Method**

This method gives a spectrophotometric method for the determination of lead.

#### **B-7.2.2 Principle**

Lead reacts with dithizone (diphenyl thiocarbazone) to form a pink coloured complex in chloroform solution. The complex is separated by extraction with chloroform from an aqueous ammonia-cyanide sulphite solution. The absorbance of the extracted complex is measured using a spectrophotometer at 510 nm wavelength.

#### **B-7.2.3 Apparatus**

**B-7.2.3.1 Standard laboratory apparatus**

**B-7.2.3.2 Separating funnels** — of 250 ml capacity.



**B-7.2.3.3 Spectrophotometer or photo colorimeter****B-7.2.4 Reagents****B-7.2.4.1 Standard lead solution**

Dissolve 0.799 2 g of A.R. lead nitrate [Pb(NO<sub>3</sub>)<sub>2</sub>] in distilled water in a beaker, transfer quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark and mix well. One millilitre of this solution contains 1 mg lead.

**B-7.2.4.1.1 Dilute standard lead solution**

Transfer 10 ml of the above solution (**B-7.2.4.1**) into a 1 litre volumetric flask, dilute to mark and mix well. One millilitre of this solution contains 10 µg of lead.

**B-7.2.4.2 Chloroform** — Reagent grade chloroform.**B-7.2.4.3 Dithizone reagent**

50 mg/l solution in chloroform. Dissolve 5 mg of the reagent in 100 ml of chloroform.

**B-7.2.4.4 Ammonia-cyanide-sulphite reagent**

To 3 ml of 10 percent potassium cyanide solution, add 35 ml of concentrated ammonia and dilute to 100 ml. Add 0.15 g of sodium sulphite and mix well.

CAUTION — POTASSIUM CYANIDE IS HIGHLY POISONOUS.

**B-7.2.4.5 Dilute hydrochloric acid solution** — Approximately 1 mol/l.**B-7.2.4.6 Hydrochloric acid solution** — Approximately 6 mol/l.**B-7.2.5 Procedure****B-7.2.5.1 Calibration**

Transfer standard lead solution (**B-7.2.4.1.1**) to a series of six 250 ml separating funnels as given below:

<i>Standard Lead Solution, ml</i>	<i>Corresponding Lead Content, µg</i>
0 (Compensation)	0
2.0	20
4.0	40
6.0	60
8.0	80
10.0	100

Add to each funnel 75 ml of ammonia-cyanide sulphite reagent solution and cautiously adjust pH to 9.5 using a pH meter (equipped with a combined electrode). This operation must be carried out slowly because, if the pH of the solution falls below 9.5 even temporarily, HCN gas may be liberated. Use of fume Cupboard during this operation is necessary. Then add 7.5 ml of dithizone reagent (**B-7.2.4.3**) to each separating funnel. Maintain time difference among each such that the time after addition of dithizone reagent and final absorbance measurement is same for all. Then add 17.5 ml of chloroform (**B-7.2.4.2**) and shake for one minute to extract the lead complex into the chloroform layer, allow the phases to separate. Transfer the chloroform layer (lower one) through a cotton plug (placed in the stem of the separating funnel) into a 1 cm cell of the spectrophotometer. Immediately replace the lid of the cell. Measure absorbance (using chloroform extract of compensation solution, in reference cell) at 510 nm wavelength.

**B-7.2.5.1.1 Calibration graph**

Plot a graph with µg of lead (in 25 ml of chloroform) as abscissae and corresponding absorbance values as ordinates.

**B-7.2.5.2 Determination**

Weigh accurately correct to potassium bromide sample 1 mg about 10 g of (the quantity to be weighed should be such that it contains 20 µg to 100 µg of lead) and transfer to 50 ml beaker, dissolve in about 20 ml to 25 ml distilled water and transfer quantitatively into a 250 ml separating funnel and rinse the beaker twice with 5 ml portions of distilled water. 75 ml of ammonia-cyanide-sulphite reagent and then by the cautious addition of dilute hydrochloric acid solution (**B-7.2.4.5**) adjust pH to 9.5. Use a pH meter with combined electrode to adjust pH. This operation must be carried out slowly because if the pH of the solution falls below 9.5 even temporarily, hydrogen cyanide (HCN) gas may be liberated. Use of fume Cupboard during this operation is necessary. Now add 7.5 ml of dithizone reagent (**B-7.2.4.3**) to the separating funnel followed by 17.5 ml dithizone reagent (**B-7.2.4.3**) to the separating funnel followed by 17.5 ml of chloroform. Shake for one minute to extract the lead into the chloroform layer. Allow the phase to separate.

Transfer the chloroform layer through a cotton plug (placed in the separating funnel stem) into a 1 cm cell of the spectrophotometer and measure the absorbance at 510 nm with chloroform in the reference cell. Read the corresponding lead content in micrograms from the calibration graph (**B-7.2.5.1.1**).

**B-7.2.5.3 Blank test**

Carry out a blank test exactly as described in (**B-7.2.5.2**) but excluding the test solution, use distilled water instead. Read the corresponding lead content in the blank from the calibration graph (**B-7.2.5.1.1**).

**B-7.2.6 Calculations and Expression of Results**

Calculate the lead content in the material from the relation given below:

$$\text{Lead content (as Pb), percent by mass} = \frac{M_1 - M_2}{M_0 \times 10\,000}$$

where

$M_1$  = mass of lead in the test solution as obtained from graph in µg,

$M_2$  = mass of lead in blank as obtained from graph in µg, and

$M_0$  = mass of sample taken for analysis in grams.

**B-7.3 Alternative Method**

Lead may alternatively be determined by ICP-OES instrumental test method as prescribed at **B-16** or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

**B-8 TEST FOR IRON**

Two methods are prescribed for determination of iron. Method A shall be the referee method and Method B the alternative method.

**B-8.1 Method A****B-8.1.1 Apparatus**

**B-8.1.1.1 Nessler cylinder** — 50 ml capacity (see IS 4161).

**B-8.1.2 Reagents**

**B-8.1.2.1 p-Nitrophenol indicator solution** — same as in **B-7.1.2**.

**B-8.1.2.2 Dilute ammonium hydroxide** — 1 : 9 (v/v).

**B-8.1.2.3 Dilute hydrochloric acid** — 1 : 99 (v/v).

**B-8.1.2.4 Acetate buffer solution**

Add 2.3 g of anhydrous sodium acetate to 58 ml of acetic acid (2 M) and dilute to 1 000 ml with water. Adjust the final pH of the solution to  $(5.0 \pm 0.1)$  with acetic acid or sodium hydroxide solution (10 percent).

**B-8.1.2.5** *o*-Phenanthroline reagent

Thoroughly mix equal volumes of *o*-phenanthroline solution (0.1 percent aqueous), hydroxylamine hydrochloride solution (10 percent aqueous) and acetate buffer solution.

**B-8.1.2.6** *Standard iron solution*

Dissolve 0.702 g of ammonium ferrous sulphate hexahydrate  $[\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}]$  in about 100 ml of water containing 10 ml of dilute sulphuric acid (4 N) and dilute to 1 000 ml. Further dilute 100 ml of the solution to 1 000 ml. One millilitre of the diluted solution contains 0.01 mg of iron (as Fe).

**B-8.1.3** *Procedure*

Dissolve 1 g of the material in 25 ml of water in a Nessler cylinder and add 1 drop of *p*-nitrophenol indicator. Add dropwise dilute ammonium hydroxide until the solution turns yellow. Add dilute hydrochloric acid, dropwise, until the solution becomes colourless and then add 2 ml in excess. Add 5 ml of *o*-phenanthroline reagent, mix well and let it stand for 10 min. Dilute to 50 ml and mix well. Simultaneously carry out a control test using 1 ml of standard iron solution for Grade 1 and 8 ml for Grade 2.

**B-8.1.3.1** 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.

**B-8.2 Method B (Colorimetric Method)****B-8.2.1** *Outline of the Method*

This standard describes a 2, 2'-bipyridyl spectrophotometric method for the determination of iron content.

**B-8.2.2** *Principle*

The dissolved iron under slightly acidic medium is reduced using hydroxyl-ammonium chloride and the colour is developed with 2,2'-bipyridyl solution [iron (II)-2,2' bipyridyl complex]. The absorbance of the colour is measured by a spectrophotometer (or equivalent calorimeter) at 522 nm.

**B-8.2.3** *Apparatus*

**B-8.2.3.1** Spectrophotometer or an equivalent photo colorimeter suitable for measurement at 522 nm.

**B-8.2.3.2** *Standard laboratory glass apparatus***B-8.2.4** *Reagents***B-8.2.4.1** *Distilled water*

**B-8.2.4.2** *Dilute hydrochloric acid* — approximately 1 mol/l.

To 910 ml of distilled water, add 90 ml of concentrated hydrochloric acid and mix.

**B-8.2.4.3** *Hydroxyl-ammonium chloride*

Dissolve 10 g of hydroxyl-ammonium chloride ( $\text{NH}_2\text{OH} \cdot \text{HCl}$ ) in water and dilute to 100 ml.

**B-8.2.4.4** *Ammonium acetate solution* — 30 g dissolved in 100 ml water.

**B-8.2.4.5** *2,2'-Bipyridyl solution*

Dissolve 1 g of 2,2'-bipyridyl in 10 ml of hydrochloric acid solution (1 mol/l) and dilute to 100 ml.

**B-8.2.4.5.1** *Standard iron solution A*

Dissolve 0.702 2 g of ferrous ammonium sulphate hexahydrate  $[\text{FeSO}_4(\text{NH}_4)_2 \cdot 6\text{H}_2\text{O}]$  in water in a suitable beaker, add 50 ml of sulphuric acid (about 1 mol/l) and mix. Transfer quantitatively into a one-litre volumetric flask, make up to the mark and mix well. One ml of this solution contains 0.1 mg iron (as Fe).

#### **B-8.2.4.5.2** *Standard iron solution B*

Dilute 100 ml of the above solution (solution A) and dilute to 1 litre in a volumetric flask, mix well. One ml of this solution contains 10  $\mu\text{g}$  of iron (as Fe).

**B-8.2.4.6** *Hydrochloric acid* — approximately 6 mol/l, (1 : 1 v/v).

#### **B-8.2.5** *Procedure*

##### **B-8.2.5.1** *Calibration*

Into each of a series of level 100 ml volumetric flask, place the quantities of standard iron solution B as given below:

<i>Standard Iron Solution B</i>	<i>Corresponding Iron Content Fe</i>
ml	$\mu\text{g}$
5.0	50
10.0	100
15.0	150
20.0	200
25.0	250
30.0	300
35.0	350
40.0	400
45.0	450
50.0	500

Add to each volumetric flask an amount of water sufficient to dilute to approximately 50 ml then 2 ml of hydrochloric acid and 2 ml of hydroxyl-ammonium chloride solution and after 5 min, add 5 ml of ammonium acetate solution and 1 ml of 2,2' bipyridyl solution. Dilute to mark, mix well, and wait for 10 minutes. Measure absorbance using spectrophotometer with 1 cm cell at 522 nm, using as reference the compensation solution.

##### **B-8.2.5.1.1** *Preparation of calibration graph*

Prepare a calibration graph with iron as Fe in  $\mu\text{g}/100\text{ ml}$  as abscissa and absorbance as ordinates.

##### **B-8.2.5.2** *Determination*

Weigh accurately to the nearest 1 mg, an amount of potassium bromide containing 10  $\mu\text{g}$  to 1 000  $\mu\text{g}$  of iron [as Fe] into a 250 ml beaker, dissolve in about 75 ml distilled water (warm on water bath if necessary) and quantitatively transfer (after cooling if warmed) into a 100 ml one-mark volumetric flask, dilute to mark with distilled water and mix well. If the solution is turbid, filter through a Whatman No. 541 filter and discard 10 ml to 15 ml of the initial filtrate.

Transfer a suitable aliquot volume (or the filtrate if filtered) containing 50  $\mu\text{g}$  to 500  $\mu\text{g}$  of Fe into a 100 ml one-mark volumetric flask. Dilute with water, add 2 ml of hydroxyl-ammonium chloride (**B-8.2.4.3**), 2 ml of hydrochloric acid (**B-8.2.4.2**), and 5 ml of ammonium acetate solution (**B-8.2.4.4**) followed by 1 ml of 2,2-bipyridyl solution and dilute to 50 ml mark, mix well and allow to stand for 10 minutes, measure the absorbance using spectrophotometer or photo colorimeter at 522 nm wavelength as described for calibration. Use the same cell size and as the one used for calibration and use blank test solution (**B-8.2.5.3**) as reference.

##### **B-8.2.5.3** *Blank*

Prepare a blank test solution using same procedure as used for determination (**B-8.2.5.2**) but excluding the potassium bromide.

#### **B-8.2.6** *Calculations and Expression of Results*

By means of the calibration graph (**B-8.2.5.1.1**) determine the mass of iron in micrograms corresponding to the absorbance value of the test solution.

Calculate the iron content as Fe in the material by the following formula:

$$\text{Iron (as Fe), percent by mass} = \frac{M_1}{M_0 \times 10\,000}$$

where

$M_1$  = mass of iron in micrograms in the test portion, and

$M_0$  = mass of the material in grams corresponding to the volume used for colour development.

#### **B-8.3** *Alternative Method*

Iron may alternatively be determined by ICP-OES instrumental test method as prescribed at **B-16** or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

#### **B-9** *TEST FOR SULPHATES*

Two methods are prescribed. Method A shall be referee method and Method B the alternative method.

##### **B-9.1** *Method A*

###### **B-9.1.1** *Apparatus*

**B-9.1.1.1** *Nessler cylinders* — 50 ml capacity (see IS 4161).

###### **B-9.1.2** *Reagents*

###### **B-9.1.2.1** *Standard sulphate solution*

Dissolve 0.148 g of anhydrous sodium sulphate in water and dilute to 1 000 ml in a volumetric flask. One millilitre of the solution contains 0.1 mg of Sulphate (as SO<sub>4</sub>)

**B-9.1.2.2** *Dilute hydrochloric acid* — 1 : 99 (v/v).

**B-9.1.2.3** *Barium chloride solution* — 10 percent.

###### **B-9.1.3** *Procedure*

Dissolve 1 g of the material in 25 ml of water and transfer to a Nessler cylinder. In another Nessler cylinder carry out a control test using 1 ml of standard sulphate solution for Grade 1 and 6 ml of standard sulphate solution for Grade 2 and 25 ml of water. To each cylinder add 6 ml of dilute hydrochloric acid and 5 ml of barium chloride solution. Dilute the contents of each cylinder to the mark and stir well.

**B-9.1.3.1** The limit prescribed in Table 1 shall be taken as not having exceeded if the turbidity produced with the material is not greater than that produced in the control test.

##### **B-9.2** *Method B*

**B-9.2.1** Turbidimetric method for the determination of sulphates is applicable when sulphate content is less than 0.1 percent by mass.

###### **B-9.2.2** *Principle*

**B-9.2.2.1** Neutralization of the test portion (for alkaline salts) with hydrochloric acid and making it acidic with slight excess of hydrochloric acid and precipitation of sulphate as barium sulphate under well-defined conditions. Measurement of turbidity using a spectrophotometer (or photo colorimeter) at 470 nm.

### **B-9.2.3** *Reagents*

#### **B-9.2.3.1** *Barium chloride*

A.R. Barium chloride dihydrate of uniform particle size between 0.50 mm and 1.25 mm, standardized by screening. It is essential that all preparations concerning the determination and standardization shall be carried out using a product of the same particle size distribution.

**B-9.2.3.2** *Sodium carbonate solution* — approximately 0.5 mol/l.

**B-9.2.3.3** *Hydrochloric acid solution* — approximately 6 mol/l.

**B-9.2.3.3.1** *Hydrochloric acid solution* — 1 mol/l

**B-9.2.3.4** *Standard sulphuric acid solution* — 0.05 mol/l.

Prepare approximately 1 mol/l solution by pouring 56 ml of concentrated sulphuric acid into 944 ml of distilled water. Dilute 50 ml of this solution to 1 000 ml in a volumetric flask which gives approximately 0.05 mol/l solution. Standardize this against a standard sodium carbonate solution (0.05 mol/l) prepared by dissolving A.R. sodium carbonate dried at 260 °C to 270 °C. Dissolve 1.324 9 g in distilled water, transfer quantitatively into a 250 ml flask, make up to mark and mix well.

**B-9.2.3.4.1** *Standard sulphuric acid solution* — 0.1 g SO<sub>4</sub>/l.

Transfer 20.8 ml of 0.05 mol/l standard sulphuric acid solution (**B-9.2.3.4**) into a 1 litre volumetric flask, dilute to mark with distilled water and mix well. One millilitre of this solution = 0.1 mg SO<sub>4</sub>.

### **B-9.2.4** *Apparatus*

**B-9.2.4.1** *Standard laboratory apparatus*

**B-9.2.4.2** *Platinum evaporating dish* — 60 ml capacity.

**B-9.2.4.3** Spectrophotometer or photoelectric absorptiometer fitted with filters giving only a negligible transmission below 450 nm and above 550 nm.

### **B-9.2.5** *Procedure*

#### **B-9.2.5.1** *Calibration*

Into each of a series of nine 50 ml one-mark volumetric flasks, place the volumes of standard sulphuric acid solution (**B-9.2.3.4**) as shown in the following table:

<i>Sulphuric Acid Solution, ml</i>	<i>Corresponding Mass of SO<sub>4</sub>, mg</i>
0 (Compensation)	0
5.0	0.5
10.0	1.0
15.0	1.5
20.0	2.0
25.0	2.5
30.0	3.0
35.0	3.5
40.0	4.0

To each flask, add 2 ml of sodium carbonate solution (**B-9.2.3.2**), and 5 ml of hydrochloric acid solution (**B-9.2.3.3.1**), stir, dilute to mark and mix well.

#### **B-9.2.5.2** *Turbidimetric reaction*

Transfer 25.0 ml of each of the solution except the first to separate dry 100 ml beaker each containing 0.15 g barium chloride (**B-9.2.3.1**). Stir by hand for 1 min at the rate of 2 revolutions per second. Allow to stand for 15 min at  $(27 \pm 2)$  °C.

NOTE — Stagger the test in order to adhere to the contact times indicated.

Stir by hand and transfer a sufficient quantity of each of the solutions to a cell of the spectrophotometer and measure the absorbance at 470 nm wavelength. Use compensation solution to adjust optical zero of spectrophotometer.

#### **B-9.2.5.3** *Preparation of calibration graph*

Plot a graph with SO<sub>4</sub> content in mg as abscissae vs corresponding absorbance values as ordinates. It should be noted that the calibration curve is linear only above 0.5 mg SO<sub>4</sub>.

#### **B-9.2.6** *Determination*

##### **B-9.2.6.1** *Test portion*

Weigh to the nearest 1 mg a quantity of the test sample of potassium bromide containing 1 mg to 3 mg of SO<sub>4</sub> and transfer into a 100 ml beaker, and dissolve in 20 ml to 25 ml distilled water, add 5 ml of hydrochloric acid solution (**B-9.2.3.3.1**) and transfer quantitatively into a 50 ml one-mark volumetric flask, make up to the mark and mix well. If turbid, filter through a Whatman No. 41 filter paper and discard a small volume of initial filtrate.

Transfer 25 ml filtrate (**B-9.2.6.1**) into a 100 ml beaker containing 0.15 g of barium chloride (**B-9.2.3.1**), stir by hand for 1 min at the rate of 2 revolutions per second. The barium chloride should then be in complete solution. Leave undisturbed for 15 min at  $(27 \pm 2)$  °C.

##### **B-9.2.6.2** *Turbidity measurement*

Transfer a sufficient quantity of the test solution (**B-9.2.6.1**) to a cell of the spectrophotometer (same size as the one used for calibration) and use to adjust optical zero of the spectrophotometer. Stir by hand the turbidity developed solution (**B-9.2.6.2**), transfer it into a cell (same size as the one used for calibration) and measure absorbance at 470 nm wavelength.

NOTE — If the turbidity developed in the test portion is beyond the limits of the calibration range, dilute an aliquot filtrate (**B-9.2.6.1**) to 50 ml and use 25 ml of this diluted solution to develop turbidity.

##### **B-9.2.6.3** *Blank test*

At the same time, carry out a blank test following the same procedure and using the same quantities of all reagents used for determination, but by replacing portion by 5 ml of standard sulphuric acid solution (**B-9.2.3.4.1**) corresponding to 0.5 mg of SO<sub>4</sub> to allow operation in the linear part of the calibration curve.

#### **B-9.2.7** *Calculation and Expression of Results*

**B-9.2.7.1** The sulphate content is obtained by the formula:

$$\text{Sulphate (as SO}_4\text{), percent by mass} = \frac{M_1 - M_2}{M_0 \times 10}$$

where

$M_1$  = mass of sulphate in milligrams corresponding to absorbance of the test portion.

$M_2$  = mass of sulphate in milligrams corresponding to the absorbance of the blank solution after deduction of 0.5 mg of SO<sub>4</sub> added (that is Graph value -0.5), and

$M_0$  = mass of the potassium bromide in grams corresponding to aliquot used for turbidity development.

**B-9.3 Alternative Method**

Sulphates may alternatively be determined by instrumental test method as prescribed at **B-17**.

**B-10 DETERMINATION OF MOISTURE****B-10.1 Procedure**

Weigh accurately about 5 g of the material in a low-form glass-stoppered weighing bottle. Dry at  $(105 \pm 2)$  °C for 4 h, cool in a desiccator and weigh to constant mass.

**B-10.2 Calculation**

$$\text{Moisture, percent by mass} = 100 \times \frac{M_1}{M_2}$$

where

$M_1$  = loss in mass in g on drying, and

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

**B-11 TEST FOR ALKALINE EARTH METALS**

**B-11.1** Two methods are prescribed for determination of alkaline earth metals. Method A shall be the referee method and Method B the alternative method.

**B-11.2 Method A****B-11.2.1 Reagents**

**B-11.2.1.1 Standard magnesium solution** — 0.02 N.

Dissolve 2.464 7 g of magnesium sulphate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) in water and dilute to 1 000 ml.

**B-11.2.1.2 Standard EDTA solution** — 0.02 N.

Weigh 3.722 g of disodium ethylenediamine tetraacetate dihydrate (EDTA) and dissolve in 500 ml of water and make up the volume to 1 000 ml. Mix thoroughly. Standardize the reagent with standard magnesium solution (**B-11.2.1.1**) using the procedure in **B-11.3**.

**B-11.2.1.3 Buffer solution** — pH 10.

Dissolve 68 g of ammonium chloride in 200 ml of water, add 570 ml of concentrated ammonia (relative density 0.91) and dilute to 1 000 ml.

**B-11.2.1.4 Rectified spirit** — see IS 323.

**B-11.2.1.5 Eriochrome black T indicator solution**

Dissolve 0.5 g of Eriochrome black T and 5 g of hydroxylamine hydrochloride in 100 ml of rectified spirit.

**B-11.2.2 Procedure**

**B-11.2.2.1** Dissolve 50 g of the material for Grade 1 and 25 g of the material for Grade 2 in 10 ml of water and make up to 1 litre. Pipette out 100 ml of the made up solution into a 250 ml conical flask, add about 25 ml of distilled water. Then add 25 ml of Buffer solution and a few drops of Eriochrome black indicator solution and titrate with EDTA solution till the colour changes from wine red to blue. Titrate further slowly near the end when a bluish green end point is reached.

**B-11.2.3 Calculation**

$$\text{Alkaline earth metals (as Mg), percent by mass} = \frac{12 \times V \times N}{W}$$

where



$V$  = volume of standard EDTA consumed in the titration,

$N$  = normality of EDTA, and

$W$  = weight of material taken for analysis.

**B-11.2.3.1** The relevant limit prescribed in Table 1 shall be taken as not having exceeded if less than 1 ml of standard EDTA solution is consumed for the test.

### **B-11.3 Method B**

#### **B-11.3.1 Apparatus**

**B-11.3.1.1** *Nessler cylinder* — 50 ml capacity (see IS 4161).

#### **B-11.3.2 Reagents**

##### **B-11.3.2.1 Standard magnesium solution**

Dissolve 1.014 g of magnesium sulphate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) in water and dilute to 1 000 ml. One millilitre of this solution contains 0.1 mg of magnesium (as Mg).

##### **B-11.3.2.2 Ammonium carbonate solution**

Dissolve 250 g of freshly powdered ammonium carbonate in a sufficient quantity of dilute ammonium hydroxide (1 : 1) to make 1 000 ml of the solution.

##### **B-11.3.2.3 Rectified spirit**

#### **B-11.3.3 Procedure**

Dissolve 10 g of the material of Grade 1 and 5 g of the material of Grade 2 in 10 ml of water in a Nessler cylinder. In another Nessler cylinder, carry out a control test by adding 5 ml of standard magnesium solution. To each cylinder add 15 ml of ammonium carbonate solution and dilute to the mark with rectified spirit. Allow to stand for 30 minutes with frequent shaking.

**B-11.3.3.1** The relevant limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced with the material is not greater than that produced in the control test.

## **B-12 DETERMINATION OF SODIUM**

### **B-12.1 Apparatus**

**B-12.1.1** *Flame Photometer* — equipped with interference filter.

NOTE — Sodium filter to be used should be of preselected or metal filter type, since ordinary sodium filters (for example, gelatine filters) have only 1 percent selectivity and work properly when the ratio of Na: K is 100:1, whereas in potassium carbonate and potassium hydroxide, the ratio is exactly reverse. Hence, results with gelatine filter are erratic.

### **B- 12.2 Reagent**

#### **B-12.2.1 Standard Sodium Solution**

Weigh exactly 2.532 g of sodium chloride, dissolve in deionized water and dilute to 1 000 ml in a measuring flask. Dilute 10 ml of this solution to 1 000 ml. One millilitre of the diluted solution contains 0.01 mg of sodium (as Na).

#### **B-12.3 Sample Solution**

Dissolve exactly 10 g of the pure grade of material (5 g in case of technical grade) in deionized water and dilute to 1 000 ml in a volumetric flask. Dilute 100 ml of this solution to 1 000 ml in a volumetric flask. Use this solution for galvanometer readings. If needed, further dilute the solution.

#### **B-12.4 Calibration Graph**

Take 10 ml, 20 ml, 40 ml, 60 ml and 80 ml of standard sodium solution in different measuring flasks and dilute to 100 ml with deionized water. Each flask now contains 0.1 mg, 0.2 mg, 0.4 mg, 0.6 mg and 0.8 mg of sodium per 100 ml.

Use these diluted solutions to obtain corresponding galvanometer reading as given in the procedure (B-12.5.2) and plot the concentration against galvanometer reading in a rectangular coordination graph. Draw a smooth curve over the points which gives a calibration graph in the range 0.1 to 1.0 percent sodium.

### B-12.5 Procedure

**B-12.5.1** Use a flame photometer equipped with atomizer, burner, optical selective device consisting of reflectors, lenses and diaphragms; and measuring instrument consisting of a photocell, an amplifier and a sensitive galvanometer. The galvanometer scale ranges from 0 to 100 divisions which measures the intensity of the radiation transmitted by the element.

**B-12.5.2** Insert the sodium filter light burner fed by illuminating gas (laboratory gas) and adjust the specified air pressure between 0.5 kg/cm<sup>2</sup> to 0.7 kg/cm<sup>2</sup> and maintain the control knob. First spray deionized water and adjust the pointer to zero in galvanometer scale by adjustment knob. Then spray the standard sodium solution and adjust the deflection to maximum (100) by using sensitivity control knob. Again spray deionized water to see that pointer comes to zero; then spray standard solution to indicate 100. Repeat till deionized water reads zero and standard solution reads 100 with the same adjustment during both the operations. Reading zero by deionized water and with the same adjustment 100 by standard solution, indicates that the instrument is now ready for measurement.

**B-12.5.3** Without altering the earlier adjustment of the instrument, spray various diluted solutions prepared in B-12.4 and obtain a calibration graph in the range 0.1 to 1.0 percent sodium. After washing with deionized water, spray the sample solution and obtain the galvanometer reading. From the graph, read out the corresponding concentration of sodium (say A).

### B-12.6 Calculation

$$\text{Sodium (as Na), percent by mass} = \frac{100 A}{M}$$

where

A = concentration of sodium corresponding to the galvanometer reading (*see* B-12.5.3), and

M = mass in g of the material taken for preparing the sample solution.

## B-13 DETERMINATION OF ARSENIC

### B-13.1 Procedure

Dissolve 1 g of the material in 10 ml of water and carry out the test for arsenic as directed in IS 2088, using 0.01 mg of arsenic trioxide for preparing the comparison stain.

### B-13.2 Alternative Method

Arsenic may alternatively be determined by ICP-OES instrumental test method as prescribed at B-16 or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

## B-14 TEST FOR BARIUM

### B-14.1 Reagents

**B-14.1.1** *Dilute Sulphuric Acid* — approximately 4 N.

**B-14.1.2** *Dilute Hydrochloric Acid* — approximately 1 N.

### B-14.2 Procedure

Dissolve 0.5 g of the material in 10 ml of water and 5 ml of 1 N hydrochloric acid and 1 ml of dilute sulphuric acid.

**B-14.2.1** The material shall be taken to have passed the test if no turbidity is produced within 5 minutes.

## B-15 DETERMINATION OF MATTER INSOLUBLE IN WATER

**B-15.1 Procedure**

Accurately weigh about 20 g of the material and dissolve it in about 150 ml of water in a beaker. Heat it on a steam bath for 30 min. Filter the solution through a tared filter paper or tared Gooch or sintered glass crucible (G No.4). Wash the residue with water. Dry the filter paper or crucible along with the residue to constant mass at  $(105 \pm 2)$  °C.

**B-15.2 Calculation**

$$\text{Matter insoluble in water, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

$M_1$  = mass in g of the residue, and

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

**B-16 DETERMINATION OF LEAD, IRON AND ARSENIC BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD****B-16.1 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-16.2 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**

(Clauses B-16.2 and B-16.4)

Sl No.	Element	Wavelength (nm)	Approximately Achievable Limits		Interfering Elements
			Radial Viewing (µg)	Axial Viewing (µg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	As	188.979	18	14	Al, Cr, Fe, Ti
		193.696	5	14	Al, Co, Fe, W, V
		197.197	(100)	31	Al, Co, Fe, Pb, Ti
ii)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-

iii)	Pb	220.353	14	5	Al, Co, Fe, Ti
		283.305	(70)		Cr, Fe
		217.00		(20)	

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**B-16.3 Reagents and Solutions****B-16.3.1 Nitric Acid (65 percent) Suprapure****B-16.3.2 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 µg/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.

**B-16.3.3 Standard Solution**

Pipette out 5 ml from 100 µg/ml standard stock solution into a 100 ml volumetric flask & 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.

**B-16.3.4 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.

**B-16.3.5 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-16.4 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-16.5 Procedure****B-16.5.1 Calibration**

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**B-16.3.5**). 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-16.5.2** 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 lead (and/or Iron, arsenic) 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-16.6 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  $\text{mg/kg}$  of the constituent elemental impurities in the sample, by multiplying the value by 20 (Dilution factor).

## **B-17 ION CHROMATOGRAPHY FOR CHLORIDES AND SULPHATES**

### **B-17.1 Principle**

Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of chlorides and sulphates. The technique separates ions and polar molecules based on their affinity to ion exchanger. When the method is employed for the determination of the anions as chlorides and sulphates, the identification should be made by using a matrix covering the ions of interest. In cation exchange chromatography, the stationary phase is functionalized with anions. These anions will attach cations towards it. These surface bound molecules/ionic species can then be removed by using a suitable eluent containing substituted ions to replace them or they can be removed by changing the  $\text{pH}$  of the column. Similarly, in anion exchange chromatography, the stationary phase is cationic in nature. These cations will then separate the anions.

Conductivity detector is generally used in this method. In case of suppressor ion exchange chromatography, analyte ions are separated on the ion exchange column and these ions together with the eluent move to the matrix suppressor. The eluent conductivity is lowered in the suppressor and the sample ion conductivity is increased leading to the large increase in signal to noise ratio.

### **B-17.2 Equipment**

**B-17.2.1 Anion Guard Column** — a protector of the separator column

**B-17.2.2 Anion Separator Column** — suitable for selective separation of ions under analysis.

**B-17.2.3 Anion Suppressor Device**

Anion micro membrane suppressor is used to analyse the data

**B-17.2.4 Detector** — conductivity detector

**B-17.2.5 Software**

Software suitable for control of various operating parameters, receiving inputs and analysis of all data

**B-17.6** Sample loop of 100  $\mu\text{l}$ , 200  $\mu\text{l}$ , 500  $\mu\text{l}$  or 1 000  $\mu\text{l}$  be used to determine ionic concentration as per instrument manual and practice.

### **B-17.3 Reagents**

**B-17.3.1 Glass or Polyethylene Sample bottles.**

**B-17.3.2 Distilled Water or Deionized Water free from the Anions of interest.**

**B-17.3.3 Eluent**

1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used. For preparation of these solution, 0.285 6 g of sodium bicarbonate and 0.381 6 g of sodium carbonate is dissolved in 2 litre of water.

**B-17.3.4 Micro Membrane Suppressor Solution: (0.025 N of sulphuric acid)**

Dilute 2.8 ml of concentrated sulphuric acid in 4 litre of water

**B-17.4 Standard solutions**

**B-17.4.1 Chloride**

Dissolve NaCl, 1.648 5 g in 1 000 ml of reagent water

**B-17.4.2 Sulphate**

Dissolve 1.81 g of potassium sulphate in 1 000 ml of reagent water

**B-17.5 Calibration and Standardization**

For each analyte of interest, prepare calibration standards at three concentration levels and a blank by adding measured stock standards and diluting with reagent water. If the concentration of the sample exceeds the calibration range, the sample may be diluted. Using 0.1 ml to 1.0 ml injections of each calibration standard, tabulate area responses or peak height against the concentration. Use these results to prepare calibration curve. Record the retention time during the procedure.

**B-17.6 Procedure**

Dissolve between 1 g to 5 g samples in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1 ml to 1.0 ml) and flush it through an injection loop using each new sample. Use the loop of same size for the standards and samples. Record the peak in size and area units. An automated constant volume injection system may preferably be used. The width of peak for retention time of ions should be same for sample and standard. Dilute the sample with the help of reagent water if the response for the peak exceeds the working range of the system for analysis. If required, spike the sample with an appropriate amount of standard and reanalyze in case of absence of distinct resolution. Retention time is inversely proportional to concentration. For solid sample of cobalt acetate, following extraction procedure may be used-

Add reagent water in an amount equal to 10 times the dry weight of the sample. The slurry is made and stirred for about 10 min using magnetic stirrer. This slurry is filtered from 0.45  $\mu$  membrane. This membrane can be directly attached to the end of the syringe. For clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no deviation from the method.

**B-17.7 Data analysis and Calculations**

Prepare a calibration curve for each analyte by plotting instrument response against concentration. Compare the sample response with the standard curve and compute sample concentration. Multiply the value by appropriate dilution factor.

Report results in mg/l or by suitably modifying into percentage. Only report those values that fall within the range of lowest and highest calibration standards.

## ANNEX C

(Clause 6)

## SAMPLING OF POTASSIUM BROMIDE

## 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** The sampling instrument and the containers for the samples shall be such that the material has no action on them. They shall be clean and dry.

**C-1.3** Samples shall be stored in brown or amber-coloured bottle.

## C-2 SCALE OF SAMPLING

## C-2.1 Lot

In a single consignment of one grade of the material, all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment of the one grade of the material is known to consist of different batches of manufacture or of different sizes of containers, then the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

**C-2.2** For ascertaining the conformity of the lot to the requirements of this specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose ( $n$ ) shall depend on the size of lot ( $N$ ) and shall be in accordance with Table 3.

**C-2.3** The containers shall be selected at random from the lot and in order to ensure randomness of selection, reference may be made to IS 4905. In case this standard is not available the following procedure is recommended for use:

Arrange all the containers in the lot in a systematic manner and starting from any container count them as 1, 2, 3... up to  $r$  and so on where  $r$  is the integral part of  $N/n$ . Every  $r^{\text{th}}$  container thus counted shall be withdrawn to give sample for tests.

Table 3 Scale of Sampling

(Clause C-2.2)

Lot Size	No. of containers to be selected
$N$	$n$
(1)	(2)
Up to 15	3
16 to 40	4
41 to 65	5
66 to 100	7
101 and above	100

NOTE — In the case of very small lots where the selection or 3 container 5 may be uneconomical, the method of judging the conformity of the lot to the requirement of the specification shall be as agreed to between the purchaser and the supplier.

## C-3 TEST SAMPLES AND REFEREE SAMPLE

**C-3.1** From each of the containers selected according to **C-2.3**, a small representative portion of the material of about 100 g shall be drawn with the help of a suitable sampling instrument.

**C-3.2** Out of these portions equal quantities of the material (not more than 90 g) shall be taken and mixed thoroughly to form a composite sample of about 250 g. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

**C-3.3** The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the  $n$  containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

**C-3.4** All the individual samples and the composite sample shall be transferred to separate sample containers. All the containers shall be sealed and labelled with full identification particulars.

**C-3.5** The referee test samples consisting of a composite sample and a set of individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of any dispute between the two.

#### **C-4 NUMBER OF TESTS**

**C-4.1** Test for the determination of potassium bromide shall be performed on each of the individual samples (*see C-3.3*).

#### **C-5 CRITERIA FOR CONFORMITY**

##### **C-5.1 For Individual Samples**

From the test results for potassium bromide, the mean ( $\bar{X}$ ) and range ( $R$ ) of test results shall be computed (range being defined as the difference between the maximum and minimum values of the test results).

**C-5.1.1** The lot shall be declared as conforming to the requirement for potassium bromide if the value of the expression ( $\bar{X} - 0.6R$ ) as calculated from the test results is equal to or greater than the corresponding value given for the relevant grade in Table 1.

##### **C-5.2 For Composite Sample**

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