

भारतीय मानक मसौदा  
पोटेशियम आयोडाइड, शुद्ध और विश्लेषणात्मक  
अभिकर्मक — विशिष्टि  
(IS 7163 का दूसरा पुनरीक्षण )

*Draft Indian Standard*  
**Potassium Iodide, Pure and Analytical Reagent—  
Specification**  
(*Second Revision of IS 7163*)

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ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01

Last date of comments: 26<sup>th</sup> March 2025

Inorganic Chemicals Sectional Committee, CHD 01

#### FOREWORD

*(Formal clauses will be added later)*

This standard was first published in 1973 and subsequently revised in 1989. The standard was revised to incorporate a new requirement of nitrogen compounds along with its method of test for the pure grade. Procedure for loss on drying was amended and Amendment No. 1 was incorporated in the first revision.

In this second revision, instrumental test methods for the determination of iron, lead, arsenic, chloride, sulphate 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 iodide is used as a reagent in analytical chemistry, in photography for precipitating silver and for medicinal purposes.

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 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 IODIDE, PURE AND ANALYTICAL  
REAGENT — SPECIFICATION***(Second Revision)***1 SCOPE**

This standard prescribes the requirements and methods of sampling and test for potassium iodide, pure and analytical reagent.

**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**

There shall be two grades of the material, namely,

- a) Pure, and;
- b) Analytical Reagent.

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

The material shall be in the form of colourless crystals, granules or powder.

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

**Table 1 Requirements for Potassium Iodide, Pure and Analytical Reagent**

*(Clause 4.2, 5.2.1, B-5.4, B-8.3.1, B-9.3.1, B-10.3.1, B-11.3.1, B-15.3.1, B-18.3.1, C-4, C-5.1.2)*

SI No.	Characteristic	Requirement for		Method of Test (Reference to Clause No. in Annex B)
		Pure	Analytical Reagent	
(1)	(2)	(3)	(4)	(5)
i)	Loss on drying, percent by mass, <i>Max</i>	1.0	0.2	<b>B-2</b>
ii)	Assay, percent by mass, <i>Min</i> (on dry basis)	99.0	99.8	<b>B-3</b>
iii)	Insoluble matter, percent by mass, <i>Max</i>	-	0.003	<b>B-4</b>
iv)	Chloride and bromide ( as Cl ), percent by mass, <i>Max</i>	-	0.01	<b>B-5</b>
v)	<i>pH</i> of 5 percent solution at 25 °C	Not more than 9.2	Between 6 and 9.2	<b>B-6</b>
vi)	Iodate (as IO <sub>3</sub> ), parts per million, <i>Max</i>	2	2	<b>B-7</b>

vii)	Phosphate (as PO <sub>4</sub> ), percent by mass <i>Max</i>	-	0.001	<b>B-8</b>
viii)	Sulphate (as SO <sub>4</sub> ), percent by mass, <i>Max</i>	0.06	0.002 5	<b>B-9</b>
ix)	Iron (as Fe), parts per million, <i>Max</i>	-	3	<b>B-10</b>
x)	Heavy metals (as Pb), percent by mass, <i>Max</i>	0.001	0.000 5	<b>B-11</b>
xi)	Sodium (as Na), percent by mass, <i>Max</i>	0.1	0.05	<b>B-12</b>
xii)	Arsenic (as As), parts per million, <i>Max</i>	2	0.1	<b>B-13</b>
xiii)	Iodine reducing substances (as I), percent by mass, <i>Max</i>	-	0.001	<b>B-14</b>
xiv)	Barium (as Ba), percent by mass, <i>Max</i>	0.001	0.001	<b>B-15</b>
xv)	Cyanide (as CN), percent by mass, <i>Max</i>	To pass the test	-	<b>B-16</b>
xvi)	Calcium group and magnesium (as Ca), percent by mass (on dry basis), <i>Max</i>	-	0.001	<b>B-17</b>
xvii)	Nitrogen compounds (as N), percent by mass, <i>Max</i>	0.001	-	<b>B-18</b>

## 5 PACKING AND MARKING

### 5.1 Packing

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

### 5.2 Marking

Each container shall be securely closed and shall bear legibly and indelibly the following information:

- a) Name and grade of the material;
- b) Name of the manufacturer and recognized trade-mark, if any;
- c) Net mass of the material; and
- d) Lot or batch number, in code or otherwise.

**5.2.1** In case of analytical reagent grade, the maximum limits for the characteristics given in Table 1 shall also be given 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.

## 6 SAMPLING

The procedure for drawing representative samples of the material and their 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 265 : 2021	Hydrochloric acid — Specification ( <i>fifth 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 3025	Methods of sampling and test (physical and chemical) for water and wastewater
(Part 2) : 2019/ISO 11885 : 2007	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> )
IS 5464: 2023	Citric acid, monohydrate — Specification ( <i>second revision</i> )

## ANNEX B

(Clause 4.2 and Table 1)

## ANALYSIS OF POTASSIUM IODIDE, PURE AND ANALYTICAL REAGENT

## B-1 QUALITY OF REAGENTS

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

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

## B-2 LOSS ON DRYING

## B-2.1 Procedure

Weigh accurately about 2 g of the finely powdered material and dry it at  $(105 \pm 2) ^\circ\text{C}$  for 4 h. Retain, the dried material for assay (*see B-3*).

## B-2.2 Calculation

$$\text{Loss on drying, percent by mass} = \frac{(M - M_1) \times 100}{M}$$

where

$M$  = mass in g, of the material taken for the test; and

$M_1$  = mass in g, of the dried material.

## B-3 ASSAY

## B-3.1 Reagents

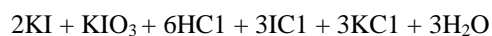
**B-3.1.1 Dilute Hydrochloric Acid** — approximately 5 N

**B-3.1.2 Chloroform****B-3.1.3 Standard Potassium Iodate Solution** — 0.05 N**B-3.1.4 Starch Indicator**

Triturate 1 g of starch with 10 ml of cold water and pour, with constant stirring, into 200 ml of boiling water. Allow to settle and use the clear supernatant liquid.

**B-3.2 Procedure**

Weigh accurately about 0.5 g of the dried material (*see B-2.1*) and dissolve in 20 ml of water in a glass stoppered flask. Add 30 ml of dilute hydrochloric acid and 5 ml of chloroform. Cool, if necessary, and titrate with standard potassium iodate solution until the iodine colour disappears from the aqueous layer. Stopper, shake vigorously for 30 s and continue the titration using starch indicator shaking vigorously after each addition of iodate solution until the iodine colour in the chloroform layer completely disappears.

**B-3.3 Calculation**

1 ml of 0.05 M  $\text{KIO}_3$  = 0.0166 g KI

$$\text{Assay, percent by mass} = \frac{1.66 \times V}{M}$$

where

$V$  = volume in ml, of standard potassium iodate solution used for the titration; and

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

**B-4 DETERMINATION OF INSOLUBLE MATTER****B-4.1 Procedure**

Dissolve 50 g of the material in 500 ml of water and heat on a steam bath for 30 min. Filter through a tared filtering crucible, wash with water and dry at  $(105 \pm 2)$  °C and weigh. Make up the volume of the filtrate to 500 ml in a measuring flask and preserve for determination of calcium group and magnesium (*see B-17*).

**B-4.2 Calculation**

$$\text{Insoluble matter, percent by mass} = \frac{100 \times M_1}{M}$$

where

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

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

**B-5 TEST FOR CHLORIDES AND BROMIDES****B-5.1 Apparatus****B-5.1.1 Nessler Cylinders** — 50 ml capacity**B-5.2 Reagents****B-5.2.1 Ammonia Solution** — 5 N**B-5.2.2 Dilute Nitric Acid** — approximately 5 N**B-5.2.3 Silver Nitrate Solution** — approximately 4 percent ( $m/v$ )**B-5.2.4 Standard Chloride Solution**

Dissolve 1.648 g of sodium chloride dried at  $(105 \pm 2) ^\circ\text{C}$ , in water and dilute to 1 000 ml. Dilute 10 ml of this solution to 100 ml. 1 ml of the diluted solution contains 0.1 mg of chloride (as Cl).

### **B-5.3 Procedure**

Weigh accurately 1 g of the material and dissolve in 5 ml of ammonia solution, add 30 ml of silver nitrate solution and 5 ml of water and shake well. Filter into a Nessler cylinder and to the filtrate, add 10 ml of dilute nitric acid and dilute to the mark. Carry out a control test using 1 ml of standard chloride solution in place of the sample and same quantities of other reagents in the same total volume of the reaction mixture.

**B-5.4** The limit prescribed in Table 1 for analytical reagent shall be taken as not having been exceeded if the turbidity produced in the test with the material, is not greater than that produced in the control test.

### **B-5.5 Alternative Method**

Chlorides may alternatively be determined by instrumental test method as prescribed at **B-20**.

## **B-6 DETERMINATION OF pH**

### **B-6.1 Apparatus**

**B-6.1.1 pH Meter** — fitted with glass electrode

### **B-6.2 Procedure**

Dissolve 10 g of the material in 200 ml of freshly boiled and cooled water and determine the pH by means of a pH meter using glass electrode at  $25 ^\circ\text{C}$ .

## **B-7 TEST FOR IODATE**

### **B-7.1 Reagents**

**B-7.1.1 Citric Acid** — see IS 5464

**B-7.1.2 Starch Solution**

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

### **B-7.2 Procedure**

Weigh accurately 1 g of the material and dissolve in 20 ml of water and add 1 g of citric acid and 1 ml of starch solution. No blue colour is produced.

## **B-8 TEST FOR PHOSPHATE**

### **B-8.1 Apparatus**

**B-8.1.1 Nessler Cylinders** — 50 ml capacity

### **B-8.2 Reagents**

**B-8.2.1 Dilute Sulphuric Acid** — approximately 5 N

**B-8.2.2 Phosphate Reagent No. 1**

Dissolve 5 g of ammonium molybdate in 100 ml of sulphuric acid (1 N)

**B-8.2.3 Phosphate Reagent No. 2**

Dissolve 0.2 g of *p*-methylaminophenol sulphate (metol) and 20 g of potassium metabisulphite in 100 ml of water.

**B-8.2.4 Standard Phosphate Solution**

Dissolve 1.43 g of potassium dihydrogen phosphate in water and make up to 1 000 ml in a measuring flask with water. Store in a polyethylene bottle. Dilute 1 ml of this phosphate solution to 100 ml in a measuring flask immediately before use. 1 ml of this diluted solution contains 0.01 mg of phosphate as  $\text{PO}_4$ .

**B-8.3 Procedure**

Dissolve 1 g of the material in 5 ml of water. Add 10 ml of nitric acid and 5 ml of hydrochloric acid and evaporate to dryness on a steam bath. Dissolve the residue in 20 ml of water. Add 3 ml of dilute sulphuric acid, 1 ml of phosphate reagent No. 1 and 1 ml of phosphate reagent No. 2, and heat over water bath at 60 °C for 10 min. Transfer the contents in a 50 ml Nessler cylinder. Do not wash. Repeat the test with 1 ml of standard phosphate solution in place of the material and take this solution in another 50 ml Nessler cylinder.

**B-8.3.1** The limit prescribed in Table 1 shall be taken as not having been exceeded if any blue colour produced with the material is not deeper than that produced with the standard solution.

**B-9 TEST FOR SULPHATES****B-9.1 General**

Two methods are prescribed for determining sulphates, namely, Method A and ion chromatography method as prescribed in **B-20**. In case of dispute, ion chromatography method shall be used as referee method.

**B-9.2 Method A****B-9.2.1 Apparatus**

**B-9.2.1.1** *Nessler cylinders* — 50 ml capacity (see IS 4161)

**B-9.2.2 Reagents**

**B-9.2.2.1** *Dilute hydrochloric acid* — 1 N

**B-9.2.2.2** *Barium chloride solution* — approximately 10 percent (m/v)

**B-9.2.2.3 Standard sulphate solution**

Dissolve 0.148 g of ignited anhydrous sodium sulphate ( $\text{Na}_2\text{SO}_4$ ) in water and dilute to 1 000 ml. 1 ml of this solution contains 0.1 mg of sulphate (as  $\text{SO}_4$ ).

**B-9.2.3 Procedure**

Dissolve 2 g of the material in 20 ml of water, add 5 ml of dilute hydrochloric acid and 2 ml of barium chloride solution. Transfer to a Nessler cylinder and dilute to the mark. Carry out a control test in another Nessler cylinder using 12 ml of standard sulphate solution for the pure grade and 0.5 ml for the analytical grade in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the turbidity after 15 ml.

**B-9.2.3.1** The limits as prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that in the control test.

**B-10 TEST FOR IRON****B-10.1 General**

Three methods are prescribed for determining iron namely, Method A, ICP-OES method as prescribed at **B-19** and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

**B-10.2 Method A****B-10.2.1 Apparatus**

**B-10.2.1.1** *Nessler cylinders* — 50 ml capacity

**B-10.2.2 Reagents**

**B-10.2.2.1** *Hydrochloric acid* — see IS 265

**B-10.2.2.2** *Ammonium persulphate*

**B-10.2.2.3** *Butanolic potassium thiocyanate solution*

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

**B-10.2.2.4** *Standard iron solution*

Dissolve 0.702 g of ammonium ferrous sulphate [FeSO<sub>4</sub>(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>·6H<sub>2</sub>O] in water containing 10 ml of dilute sulphuric acid and dilute with water to 1 000 ml. Dilute 10 ml of the solution to 100 ml. 1 ml of the dilute solution contains 0.01 mg of iron (as Fe).

**B-10.2.3** *Procedure*

Dissolve 2 g of the material in 10 ml of water and 10 ml of hydrochloric acid evaporate to dryness on a steam bath. Add 5 ml of hydrochloric acid and again evaporate to dryness. Prepare a control test by adding 0.6 ml of standard iron solution and 15 ml of hydrochloric acid and evaporate to dryness on steam bath. Take up each of the residues separately in 2 ml of hydrochloric acid, contained in Nessler cylinders and add about 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Shake vigorously for 30 s and allow to settle. Compare the colour of the solution in two cylinders.

**B-10.2.3.1** The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the red colour produced in the test is not greater than that produced in the control test.

**B-11 TEST FOR HEAVY METALS (as Pb)****B-11.1** *General*

Three methods are prescribed for determining heavy metals (as Pb), namely, Method A, ICP-OES method as prescribed at **B-19** and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

**B-11.2** *Method A***B-11.2.1** *Apparatus*

**B-11.2.1.1** *Nessler cylinder* — 50 ml capacity

**B-11.2.2** *Reagents*

**B-11.2.2.1** *p-Nitrophenol indicator solution* — dissolve 0.2 g of *p*-nitrophenol in hot water and dilute to 100 ml

**B-11.2.2.2** *Dilute ammonium hydroxide* — 1: 9 (v/v)

**B-11.2.2.3** *Dilute hydrochloric acid* — approximately 0.1 N

**B-11.2.2.4** *Hydrogen sulphide solution* — saturated and freshly prepared

**B-11.2.2.5** *Standard lead solution*

Dissolve 1.60 g of lead nitrate in water and dilute to 1 000 ml. Dilute 10 ml of the solution to 1 000 ml. 1 ml of this solution contains 0.01 mg of lead (as Pb).

**B-11.2.3** *Procedure*

Weigh 1 g of the material for pure grade and 2 g for analytical reagent grade and dissolve in 25 ml of water in a Nessler cylinder and add one drop of *p*-nitrophenol indicator solution. Add dilute ammonium hydroxide dropwise 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 in place of the material.

**B-11.2.3.1** The relevant 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 produced in the control test.

**B-12 TEST FOR SODIUM****B-12.1** *Apparatus*

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



**B-12.2 Reagents****B-12.2.1 Standard Sodium Solution**

Weigh exactly 2.542 g of sodium chloride, dissolve in deionized water and dilute to 1 litre with deionized water in a measuring flask. The solution contains 0.1 g of sodium (as Na) per 100 ml of standard sodium solution.

**B-12.2.2 Calibration Graph**

Take 10, 20, 40, 60 and 80 ml of standard sodium solution (*see* **B-12.2.1**) and dilute to 100 ml with deionized water in different measuring flasks. Each flask now contains 0.01, 0.02, 0.04, 0.06 and 0.08 g of sodium per 100 ml of the solution. Use these dilute solutions to obtain a corresponding galvanometer reading as described in **B-12.3** 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 of 0.01 g to 0.1 g of sodium per 100 ml of the standard sodium solution.

**B-12.2.3 Sample Solution**

Dissolve an appropriate quantity of the sample in minimum quantity of deionized water and dilute to 100 ml in measuring flask with deionized water so as to give sodium concentration between 0.01 and 0.1 g per 100 ml of the solution.

**B-12.3 Procedure**

**B-12.3.1** Use a flame photometer equipped with atomizer and burner; optical selective device consisting of reflectors, lenses and diaphragms; and measuring instrument consisting of photocell, amplifier and sensitive galvanometer. The galvanometer scale ranges from 0 to 100 divisions and it measures the intensity of the radiation transmitted by the element.

**B-12.3.2** Insert the sodium filter corresponding to wavelength 589 nm, light the burner fed by illuminating gas (laboratory gas) and adjust the specific air pressure between 0.5 and 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 zero adjustment knob, then spray the standard sodium solution (**B-12.2.1**) and adjust the deflection to the maximum (100) by using sensitivity control knob. Again spray water to see that the pointer comes to zero and then spray standard solution to indicate 100. Repeat till water reads zero and standard solution reads 100 with the same adjustment during both operations which indicates that the instrument is now ready for measurement.

**B-12.3.3** Without altering the earlier adjustment of the instrument, spray various diluted solutions prepared in **B-12.2.2** and obtain a calibration graph in the range 0.01 to 0.1 g of sodium per 100 ml of the solution. After washing with deionized water, spray the sample solution and obtain the galvanometer reading. From the graph, read out the corresponding concentration of sodium in the solution (say, A).

**B-12.4 Calculation**

$$\text{Sodium, percent by mass} = 100 \times \frac{A}{M}$$

where

$A$  = concentration of sodium corresponding to the galvanometer reading (*see* **B-12.3.3**); and  
 $M$  = mass in g, of the dried material taken for preparing the sample solution.

**B-13 TEST FOR ARSENIC****B-13.1 General**

Three methods are prescribed for determining arsenic, namely, Method A, ICP-OES method as prescribed at **B-19** and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

**B-13.2 Method A**

Dissolve 1 g of the material in 10 ml of water and carry out the test for arsenic as prescribed in IS 2088 using 0.002 64 mg for pure grade and 0.000 132 mg for analytical grade of arsenic trioxide ( $\text{As}_2\text{O}_3$ ) for preparing the comparison stain.

## **B-14 DETERMINATION OF IODINE REDUCING SUBSTANCES**

### **B-14.1 Reagents**

**B-14.1.1** *Dilute Sulphuric Acid* — approximately 5 N

**B-14.1.2** *Starch Indicator Solution*

**B-14.1.3** *Iodine Solution* — 0.002 N

### **B-14.2 Procedure**

Weigh accurately 1.5 g of the material and dissolve in 20 ml of freshly boiled and cooled water and add 2 ml of dilute sulphuric acid, 1 ml of starch indicator solution and 0.05 ml of iodine solution.

**B-14.2.1** The material shall be regarded to have passed the requirement of the test if a blue colour is produced and the colour persists for 30 s.

## **B-15 TEST FOR BARIUM**

### **B-15.1 Apparatus**

**B-15.1.1** *Nessler Cylinders* — 100 ml capacity

### **B-15.2 Reagents**

**B-15.2.1** *Dilute Hydrochloric Acid* — 1 N

**B-15.2.2** *Barium Chloride Solution* — approximately 10 percent (*m/v*)

**B-15.2.3** *Dilute Sulphuric Acid* — 5 N

**B-15.2.4** *Rectified Spirit* (*see* IS 323)

**B-15.2.5** *Standard Sulphate Solution*

Dissolve 0.148 g of ignited anhydrous sodium sulphate ( $\text{Na}_2\text{SO}_4$ ) in water and dilute to 1 000 ml. 1 ml of this solution contains 0.1 mg of sulphate (as  $\text{SO}_4$ ).

**B-15.2.6** *Sodium Thiosulphate Solution* — approximately 0.1 N

### **B-15.3 Procedure**

Dissolve 7 g of the material in 40 ml of water and add 10 ml of rectified spirit; mix, add 0.5 ml of dilute sulphuric acid, mix immediately and set aside for 1 h. Discharge any colour due to liberated iodine by the dropwise addition of 0.1 N sodium thiosulphate solution. To another Nessler cylinder, add 0.5 ml of standard sulphate solution, 37 ml of water, 10 ml of rectified spirit and 1 ml of dilute hydrochloric acid, mix and add 1 ml of barium chloride solution, mix immediately and allow to stand for 1 h.

**B-15.3.1** The limits 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 with the standard solution.

## **B-16 TEST FOR CYANIDE**

### **B-16.1 Apparatus**

**B-16.1.1** *Nessler Cylinder or Test Tube* — 50 ml capacity

### **B-16.2 Reagents**

**B-16.2.1** *Ferrous Sulphate* — saturated solution

**B-16.2.2** *Dilute Hydrochloric Acid* — 1 N

**B-16.2.3** *Sodium Hydroxide Solution* — 10 percent (*m/v*)

**B-16.3 Procedure**

Dissolve 0.5 g of the material in 5 ml of warm water contained in a Nessler cylinder. Add 1 drop of ferrous sulphate solution and 0.5 ml of sodium hydroxide solution. Acidify the contents with dilute hydrochloric acid.

**B-16.3.1** The material shall be taken to have passed the test if no blue colour is produced in the Nessler cylinder.

**B-17 DETERMINATION OF CALCIUM GROUP AND MAGNESIUM****B-17.1 Reagents****B-17.1.1 Standard Calcium Solution**

Weigh 1 g of calcium carbonate dried at 120 °C and dissolve it in the minimum quantity of dilute hydrochloric acid. Dilute the solution to 1 litre in a graduated flask. 1 ml of the solution is equivalent to 0.400 8 mg of calcium (as Ca).

**B-17.1.2 Standard EDTA Solution**

Dissolve 3.72 g of disodium ethylene diamine tetracetate dihydrate in water and dilute in a graduated flask to 1 litre. The solution shall be standardized frequently against the standard calcium solution, following the procedure given in **B-17.2**.

**B-17.1.3 Eriochrome Black T Indicator Solution**

Dissolve 0.1 g of the dye in 20 ml of rectified spirit (*see* IS 323). The solution shall be prepared fresh every week.

**B-17.1.4 Ammonium Chloride — Ammonium Hydroxide Buffer Solution**

Dissolve 67.5 g of ammonium chloride in a mixture of 570 ml of ammonium hydroxide (sp gr 0.90) and 250 ml of water. Also dissolve separately a mixture of 0.931 g of disodium ethylene diamine tetracetate dihydrate and 9.616 g of magnesium sulphate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) in about 50 ml of water. Mix the two solutions and dilute to 1 litre.

NOTE — 5 ml of the buffer solution added to 50 ml of water should not consume more than a drop of EDTA solution to change to distinct blue with eriochrome black T indicator.

**B-17.2 Procedure****B-17.2.1 Standardization of EDTA Solution**

Transfer 25 ml of standard calcium solution into a conical flask, add 25 ml of water, 10 ml of ammonium chloride-ammonium hydroxide buffer solution, 5 drops of the eriochrome black T indicator solution and titrate against the standard EDTA solution to a clear blue end point.

**B-17.2.2** Titrate 25 ml of the buffer solution with EDTA solution using eriochrome black T indicator. Subtract the buffer correction for 10 ml (usually it will be 0.1 ml) from the reading obtained in **B-17.2.1** and note the final titre value. Calculate the calcium equivalent of 1 ml of EDTA solution (say, *A*).

**B-17.2.3** Transfer exactly 100 ml of the solution preserved in **B-4.1** into a 250 ml conical flask. Add 20 ml of ammonium chloride ammonium hydroxide buffer solution, 5 drops of eriochrome black T indicator solution and titrate against standard EDTA solution till wine red colour of the solution changes to pure blue end point. Note the volume of EDTA solution used in the titration.

**B-17.3 Calculation**

$$\text{Calcium group and magnesium (as Ca), percent by mass (on dry basis)} = \frac{A \times V_1}{2 \times M}$$

where

*A* = calcium equivalent in mg of 1 ml of EDTA solution determined in **B-17.2.2**;

*V*<sub>1</sub> = volume in ml, of standard EDTA solution used in **B-17.2.3**; and

*M* = mass in g, of the dried sample taken for the test.

**B-18 TEST FOR NITROGEN COMPOUNDS****B-18.1 Apparatus**

**B-18.1.1 Nessler Cylinder** — 50 ml capacity (*see* IS 4161)

**B-18.2 Reagents****B-18.2.1 Dilute Hydrochloric Acid** — approximately**B-18.2.2 Sodium Hydroxide Solution** — 10 percent**B-18.2.3 Aluminum Wire****B-18.2.4 Nessler Reagent**

Dissolve 10 g of potassium iodide in 10 ml of ammonia free water, and add to it slowly, while stirring, saturated mercuric chloride solution until a slight permanent precipitate is formed. Add 30 g of potassium hydroxide and, when it has dissolved, add 1 ml of mercuric chloride solution and dilute to 200 ml with ammonia free water. Allow to settle overnight, decant the clear solution and keep it in a bottle closed with a well-fitting rubber stopper.

**B-18.2.5 Standard Nitrogen Solution**

Dissolve 0.3819 g of ammonium chloride in enough water to make up the volume to 1 000 ml. Pipette out 10 ml from this solution and dilute with water to 100 ml. 1 ml of this solution is equivalent to 0.01 mg of nitrogen.

**B-18.3 Procedure**

Dissolve 1 g of the material in 60 ml of water in a flask connected through a spray trap to a condenser, the end of which dips beneath the surface of 10 ml of dilute hydrochloric acid. Add to the solution in the flask, 20 ml of freshly boiled sodium hydroxide solution and about 0.5 g of aluminum wire in small pieces. Allow to stand for 1 h and distill over about 35 ml. Transfer the distillate into a Nessler cylinder, add 1 ml of sodium hydroxide solution, dilute to 50 ml and add 2 ml of Nessler reagent. Carry out a control test in another Nessler cylinder in the same manner using 1 ml of standard nitrogen solution in place of the material.

**B-18.3.1** The material shall be taken as not having exceeded the limit given in Table 1 if the colour produced is not darker than that produced in the control test.

**B-19 DETERMINATION OF ARSENIC, IRON AND LEAD BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD****B-19.1 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-19.2 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-19.2 and B-19.4)

SI No.	Element	Wavelength (nm)	Approximately Achievable Limits		Interfering Elements
			Radial Viewing	Axial Viewing	

(1)	(2)	(3)	( $\mu\text{g}$ ) (4)	( $\mu\text{g}$ ) (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)	

**B-19.3 Reagents and Solutions****B-19.3.1. Nitric Acid (65 percent) Suprapure****B-19.3.2 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.

**B-19.3.3 Standard Solution**

Pipette out 5 ml from 100  $\mu\text{g}/\text{ml}$  standard stock solution into a 100 ml volumetric flask and make up volume with 2 percent nitric acid to prepare 5  $\mu\text{g}/\text{ml}$  solution. From this 5  $\mu\text{g}/\text{ml}$  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  $\mu\text{g}/\text{ml}$  solution of respective elements under reference.

**B-19.3.4 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-19.3.5 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-19.4 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-19.5 Procedure****B-19.5.1 Calibration**

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**B-19.3.5**). 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-19.5.2** 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  $\pm 5$  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-19.6 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).

**B-20 ION CHROMATOGRAPHY FOR CHLORIDES AND SULPHATES****B-20.1 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  $pH$  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-20.2 Equipment**

**B-20.2.1 Anion Guard Column** — a protector of the separator column

**B-20.2.2 Anion Separator Column** — suitable for selective separation of ions under analysis.

**B-20.2.3 Anion Suppressor device**

Anion micro membrane suppressor is used to analyse the data

**B-20.2.4 Detector** — conductivity detector

**B-20.2.5 Software**

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

**B-20.2.6** Sample loop of 100 µl, 200 µl, 500 µl or 1000 µl be used to determine ionic concentration as per instrument manual and practice.

**B-20.3 Reagents**

**B-20.3.1** *Glass or Polyethylene Sample bottles.*

**B-20.3.2** *Distilled Water or Deionized Water free from the Anions of interest.*

**B-20.3.3 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-20.3.4** *Micro Membrane Suppressor Solution: (0.025 N of sulphuric acid)*

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

**B-20.4 Standard solutions****B-20.4.1 Chloride**

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

**B-20.4.2 Sulphate**

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

**B-20.5 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-20.6 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 µ 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-20.7 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 IODIDE, PURE AND ANALYTICAL REAGENT

#### C-1 GENERAL REQUIREMENTS OF SAMPLING

**C-1.1** In drawing samples, the following pre-cautions and directions shall be observed.

**C-1.2** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

**C-1.3** The samples shall be placed in suitable, clean and dry containers.

**C-1.4** The sample containers shall be of such a size that they are almost completely filled by the sample.

**C-1.5** Each sample container shall be sealed airtight after filling and marked with full details of sampling and the date of sampling.

#### C-2 SCALE OF SAMPLING

##### C-2.1 Lot

All the containers in a single consignment of the material of same grade and drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

**C-2.1.1** Samples shall be tested for each lot for ascertaining conformity of the material to the requirements of the specification. **C-2.2.** The number ( $n$ ) of containers to be selected from the lot shall depend on the size of the lot ( $N$ ) and shall be in accordance with Table 3.

**Table 3 Number of Containers to be selected for Sampling**

(Clause C-2.1.1)

Sl. No.	Lot Size	No. of Containers to be Selected	
		$N$	$N$
(1)	(2)	(3)	
i)	Up to 25		3
ii)	26 to 50		4
iii)	51 to 100		5
iv)	101 and above		6

NOTE — When the size of the lot is 3 or less, the number of containers to be selected and the criterion for judging the conformity of the lot to the specification shall be as agreed to between the purchaser and the supplier.

**C-2.3** These containers shall be selected at random from the lot and in order to ensure randomness of selection, a random number table as agreed to between the purchaser and the supplier shall be used. In case such a table is not available, the following procedure shall be adopted.

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



**C-3 PREPARATION OF TEST SAMPLES AND REFEREE SAMPLE**

**C-3.1** For drawing samples a ladle or any other suitable instrument shall be used.

**C-3.2** In the case of large containers, such as, drums, sample shall be collected from different depths with the help of the sampling instrument. In the case of small containers, the contents shall be thoroughly mixed before taking the sample from them.

**C-3.3** Out of these portions collected from all the selected containers, an equal quantity of the material shall be taken and mixed together to form a composite sample of about 300 g. The composite sample so formed 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-3.4** The remaining portions of the material from each container shall be divided into three equal parts and each such separate part shall constitute an individual sample. One set of individual samples (representing the  $n$  containers selected) shall be marked for the purchaser, another for the supplier and the third for the referee.

**C-3.5** All the individual and composite samples shall be immediately transferred to separate glass bottles and labelled with full identification particulars on the bottles.

**C-3.6** The referee sample shall bear the seal of both the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier to be used in case of dispute between the two.

**C-4 NUMBER OF TESTS**

Tests for potassium iodide shall be conducted on each individual sample and tests for all other characteristics given in 3 and Table 1 shall be conducted on the composite samples.

**C-5 CRITERIA FOR CONFORMITY**

**C-5.1** A lot shall be declared as conforming to the requirements of this specification if the stipulations laid down in **C-5.1.1** and **C-5.1.2** are fulfilled.

**C-5.1.1 Assay**

From the individual test results for assay, the mean ( $\bar{x}$ ) and the range ( $R$ ) of the test results shall be calculated (range being the difference between the maximum and the minimum test results). The value of the expression  $(\bar{x} - 0.6 R)$  shall be greater than or equal to 99.0 for pure grade and 99.8 for analytical reagent grade.

**C-5.1.2 For Other Characteristics**

The test results for all other characteristics prescribed in 3 and Table 1 shall satisfy the relevant requirements.