भारतीय मानक मसौदा

पोटैशियम आयोडेट — विशिष्टि

(*IS 13057 का पहला पुनरीक्षण*)

Draft Indian Standard

Potassium Iodate — Specification

(*First Revision of IS 13057*)

ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01	**Last date of comments: 9th December 2024**

Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

(*Formal clauses will be added later*)

This standard was first published in 1991. In this first revision, instrumental test methods for the determination of chloride, sulphate, lead, arsenic and iron 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 iodate is widely used for iodization of edible common salt. It is used as feed additive and as maturing agent and dough conditioner. It is also used in iodometry and medicines. In this standard three grades of potassium iodate, namely, analytical, food and technical grades, have been covered.

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 IODATE — SPECIFICATION

(*First Revision*)

1 SCOPE

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

2 REFERENCES

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

IS No	*Title*
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 2088 : 2023	Methods for determination of arsenic (*third revision*)
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) (*first revision*)
(Part 65) : 2022/ ISO 17294-2 : 2016	Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including uranium isotopes (*first revision*)
IS 4905 : 2015 ISO 24153 : 2009	Random sampling and randomization procedures (*first revision*)

3 GRADES

There shall be three grades of the material, namely:

a) Analytical Reagent (AR);
b) Food (FD); and
c) Technical (Tech).

4 REQUIREMENTS

4.1 Description

The material shall be in the form of white crystalline powder, free from impurities.

4.2 The material shall comply with the requirements given in Table 1 when tested in accordance with the relevant test methods prescribed in Annex A.

5 PACKING AND MARKING

5.1 Packing

5.1.1 The material shall be packed in airtight bottles of capacity 500 g, 1 kg and 2.5 kg.

5.1.2 For bulk packaging, the material shall be packed in steel or fiber drums having polyethylene liner bags, with capacity of 10 kg, 25 kg and 50 kg.

5.2 Marking

5.2.1 The containers shall be securely closed and marked legibly and indelibly with the following information:

a) Name of the material;
b) Mass of the material in the package;
c) Indication of the source of manufacture; and
d) Lot or batch number, in code or otherwise.

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 test samples of the material and the criteria for conformity shall be as prescribed in Annex B.

Table 1 Requirements for Potassium Iodate

(*Clauses* 4.2, B-4, B-5.1 and B-5.2)

SL No	**Characteristic**	**Requirement**			**Method of Test (Ref to Cl No. in Annex A)**
		AR Grade	FD Grade	Tech Grade	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Potassium iodate (as KIO_3), percent by mass, *Min*	99.9	99.0	99.0	**A-3**
ii)	Matter insoluble in water, percent by mass, *Max*	0.003	—	—	**A-4**
iii)	Free acid, percent by mass, *Max*	0.002	—	—	**A-5**
iv)	Free alkali, percent by mass, *Max*	0.002	—	—	**A-5**
v)	Bromate, bromide, chlorate and chloride (as Cl), percent by mass, *Max*	0.02	0.01	0.1	**A-6 or A-16**
vi)	Iodide (as I), percent by mass, *Max*	0.001	0.002	0.005	**A-7**
vii)	Nitrogen compound (as N), percent by mass , *Max*	0.002	—	—	**A-8**
viii)	Sulphate (as SO_4), percent by mass, *Max*	0.01	—	0.1	**A-9 or A-16**
ix)	Iron (as Fe), percent by mass, *Max*	0.001	—	—	**A-10 or A-17**
x)	Heavy metals (as Pb), ppm, *Max*	10	10	—	**A-11 or A-17**
xi)	Sodium (as Na), percent by mass, *Max*	0.01	—	0.05	**A-12**
xii)	Loss on drying, percent by mass, *Max*	0.05	0.5	—	**A-13**
xiii)	Arsenic (as As), ppm, *Max*	—	3	—	**A-14 or A-17**
xiv)	*p*H (5 percent solution)	5.5 to 9.0	5.5 to 9.0	5.5 to 9.0	**A-15**

ANNEX A

(*Clause* 4.2)

METHODS OF TEST FOR POTASSIUM IODATE

A-1 QUALITY OF REAGENTS

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.

A-2 PREPARATION OF SAMPLE

A-2.1 Sample for determination of potassium iodate content shall be the residual material obtained at the end of the test for loss on drying.

A-2.2 Sample for Other Tests

Crush 100 g of the sample so that it passes through 1 mm IS Sieve, and keep it in a clean glass-stoppered weighing bottle in a desiccator for subsequent tests.

A-3 DETERMINATION OF POTASSIUM IODATE

A-3.1 Reagents

A-3.1.1 *Potassium Iodide*

A-3.1.2 *Hydrochloric Acid* **—** approximately 5 N**.**

A-3.1.3 *Standard Sodium Thiosulphate Solution* **—** 0.1 N.

A-3.1.4 *Starch Indicator Solution*

Mix 0.5 g of soluble starch with 5 ml of cold water. Add this mixture, with stirring, to 95 ml of boiling water and heat in a boiling water bath for 5 min.

A-3.2 Procedure

Dissolve 1.5 g of the dried material (at 130 °C) in water and dilute to 500 ml. To 50 ml of this solution add 3 g of potassium iodide and 10 ml of dilute hydrochloric acid and titrate the liberated iodine with 0.1 N standard sodium thiosulphate solution, using starch as indicator. Perform a blank determination and make necessary correction, if any.

A-3.3 Calculation

$$\text{Potassium iodate (as } KIO_3\text{,) percent by mass} = \frac{3.567\,(V_1 - V_2)}{M}$$

where

V_1 = volume in ml of standard sodium thiosulphate solution used in titrating the sample,
V_2 = volume in ml of standard sodium thiosulphate solution used in titrating the blank, and
M = mass in g of the sample taken for the test

A-4 DETERMINATION OF MATTER INSOLUBLE IN WATER

A-4.1 Procedure

Dissolve 20 g of the sample (*see* **A-2.2**) in about 400 ml of warm water. Cool the solution and set it aside for 1 h. Filter through a weighed sintered glass crucible (G No. 4). Wash the residue with water and dry to constant mass a 105 °C.

A-4.2 Calculation

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

where

M_1 = mass in g of the residue, and
M_2 = mass in g of the sample taken for the test.

A-5 TEST FOR FREE ACID AND FREE ALKALI

A-5.1 Apparatus

A-5.1.1 *p*H Meter

A-5.2 Reagents

A-5.2.1 *Sodium Hydroxide Solution* — 0.02 N.

A-5.2.2 *Hydrochloric Acid Solution* — 0.02 N.

A-5.3 Procedure

Dissolve 10 g of the sample in 200 ml of carbon dioxide free water and titrate with sodium hydroxide or hydrochloric acid to *p*H 7.0. Not more than 0.25 ml of sodium hydroxide or hydrochloric acid shall be required.

A-6 TEST FOR BROMATE, BROMIDE, CHLORATE AND CHLORIDE

A-6.1 Reagents

A-6.1.1 *Sulphur Dioxide*

A-6.1.2 *Dilute Ammonia Solution*

Approximately 5 N. Add 280 ml of strong ammonia solution (AR or GR grade, rd 0.88) to sufficient water to make up the volume to 1 000 ml.

A-6.1.3 *Silver Nitrate Solution* — 0.1 N.

Dissolve 16.087 g of silver nitrate (AR or GR grade) in sufficient water to make up the volume to 1000 ml.

A-6.1.4 *Dilute Nitric Acid* — approximately 5 N.

Add 320 ml of nitric acid (AR or GR grade, rd 1.42) to sufficient water and make up the volume to 1000 ml.

A-6.1.5 *Standard Chloride Solution*

Dissolve 0.164 g of sodium chloride (NaCl) in water and dilute to 1 000 ml. One milliliter of this solution contains 0.1 mg of chloride (as Cl).

A-6.2 Procedure

A-6.2.1 Dissolve 0.5 g of the sample in 50 ml of water and pass sulphur dioxide into the solution until the precipitate of iodine dissolves. Evaporate the solution to about 2 ml, cool, add 6 ml of dilute ammonia solution, transfer to a stoppered test tube and dilute to 15 ml with water. Add 15 ml of silver nitrate solution, shake well and filter. To 15 ml of filtrate, add 30 ml of water and 5 ml of dilute nitric acid. Any opalescence produced shall not be greater than the standard opalescence produced as in **A-6.2.2.**

A-6.2.2 *Standard Opalescence*

To 50 ml of water add the following quantity of standard chloride solution, 1 ml of dilute nitric acid and 1 ml of silver nitrate solution, mixing after each addition:

Grade of Material	Quantity of Standard Chloride Solution

	ml
AR	1
FD	0.5
Tech	5

Use as comparison standard after 5 min.

A-6.3 Alternative Method

Chlorides may alternatively be determined by instrumental test method as prescribed at **A-16**.

A-7 TEST FOR IODIDE

A-7.1 Reagents

A-7.1.1 *Dilute Sulphuric Acid*

A-7.1.2 *Starch Indicator Solution*

Triturate 1 g of arrowroot starch with 10 ml of cold water and pour slowly, with constant stirring, into 200 ml of boiling water. Boil the mixture until a thin translucent fluid is obtained. Allow to settle and use only the clear, supernatant liquid. Prepare this solution fresh.

A-7.2 Procedure

For AR grade, dissolve 2 g of the material in 10 ml of water and add 1 ml of dilute sulphuric acid and 1 drop of starch solution. No blue Colour shall be formed. Use 1 g of the material for testing food grade and 0.4 g of the material for testing technical grade of potassium iodate.

A-8 TEST FOR NITROGEN COMPOUNDS

A-8.1 Apparatus

Micro Kjeldahl's Distillation Apparatus

A-8.2 Reagents

A-8.2.1 *Sodium Hydroxide Solution* (*Nitrogen Free*).

To 50 ml of sodium hydroxide solution (approximately 5 N) add 100 ml of water and 0.1 g of aluminum wire and boil down to 50 ml. Cool and decant from any residue.

A-8.2.2 *Sodium Hydroxide Solution* (*Ammonia Free*)

To 50 ml of sodium hydroxide solution (approximately 5 N) add 100 ml of water and boil to reduce the volume to 50 ml.

A-8.2.3 *Aluminum Wire*

A-8.2.4 *Hydrochloric Acid* — 0.1 N.

A-8.2.5 *Standard Ammonium Solution*

Dissolve 2.97 g of ammonium chloride (AR or GR grade) in sufficient water and make it to 100 ml. (This solution shall be freshly prepared.) Dilute 1 ml of this solution to 100 ml with water immediately before use. One milliliter of the standard ammonium solution thus prepared contains 0.01 mg of NH_4.

A-8.2.6 *Nessler's Reagent*

Dissolve 35 g of potassium iodide (AR or GR grade) and 12.5 g of mercuric chloride (AR or GR grade) in 800 ml of water and add cold saturated solution of mercuric chloride until a slight permanent precipitate is produced. Then add 120 g of sodium hydroxide (AR or GR grade).

Dissolve and add a little more of mercuric chloride solution and sufficient water to make the volume to 1000 ml. Shake occasionally for several days and allow to settle and decant the clear liquid.

A-8.3 Procedure

To 1 g of the sample in micro-Kjeldahl's distillation apparatus add 25 ml of sodium hydroxide solution (nitrogen free) and 1 g of aluminum wire. Pass steam into the distillation flask and collect any distillate in 5 ml of water containing 1 ml of hydrochloric acid. Continue to pass steam till the liquid boils. Disconnect the steam, allow the reaction to proceed for 1 h and then steam distil until a total volume of 50 ml is obtained during about 6 min. Prepare a standard in the same manner using 2.58 ml of standard ammonium solution in place of the sample.

To 25 ml of each distillate add 25 ml of water, 2 ml of sodium hydroxide solution (ammonia free) and 2 ml of Nessler's reagent. Any yellow Colour produced in the test shall not be deeper than that produced in the standard.

A-9 TEST FOR SULPHATE

A-9.1 Method A (Turbidimetric Method)

A-9.1.1 *Reagents*

A-9.1.1.1 *Industrial Methylated spirit* (*absolute*, 74 *OP*)

A-9.1.1.2 *Industrial methylated spirit* (20 *percent*)

Dilute 20 ml of absolute industrial methylated spirit with water to produce a final volume of 100 ml.

A-9.1.1.3 *Barium chloride solution* — approximately 1 N.

Dissolve 122 g barium chloride dehydrate in water and make up the volume to 1 000 ml.

A-9.1.1.4 *Dilute hydrochloric acid* — approximately 5 N.

Dilute 430 ml of hydrochloric acid (AR or GR grade rd 1.18) with sufficient water to make up the volume to 1 000 ml.

A-9.1.1.5 *Standard sulphate solution*

Dilute 20.8 ml of 0.1 N sulphuric acid with sufficient water to make up the volume to 1 000 ml. This solution contains 0.1 mg of SO_4 per ml.

A-9.1.2 *Procedure*

A-9.1.2.1 Dissolve 1 g of the sample in 40 ml of water, add 0.6 g of hydroxyl ammonium chloride and boil till the liberated iodine is expelled. Cool, dilute to 40 ml with water, add 10 ml of absolute industrial methylated spirit and 1 ml of dilute hydrochloric acid. Mix the resulting solution after adding 1 ml of barium chloride solution, and allow to stand for 1 h. Any turbidity produced shall not be greater than the standard turbidity produced as in **A-9.1.2.2**.

A-9.1.2.2 *Standard turbidity*

Take 1 ml of standard sulphate solution for AR grade and 10 ml for Tech grade of the material. Add 37 ml of water, 10 ml of absolute industrial methylated spirit, 1 ml of dilute hydrochloric acid and mix. Add 1 ml of barium chloride solution mix immediately and allow to stand for 1 h.

A-9.2 Method B (Gravimetric Method)

A-9.2.1 *Reagents*

A-9.2.1.1 *Dilute hydrochloric acid* — approximately 4 N.

A-9.2.1.2 *Barium chloride* — approximately 10 percent.

A-9.2.2 Procedure

Dissolve 1 g of the sample in 40 ml of water, add 0.6 g of hydroxyl ammonium chloride and boil till the liberated iodine is expelled. Cool and dilute to 400 ml with water. Add one drop of methyl orange and about 10 ml of dilute hydrochloric acid till it becomes pink and then boil. To the boiling solution add barium chloride solution drop by drop so that the addition is in slight excess and continue boiling for 4 min to obtain a granular precipitate. Allow it to stand for 4 h and filter through a weighed sintered glass crucible (G No. 4). Wash the precipitate till it is free from chloride and dry to constant mass at 105 °C to 110 °C. Alternatively, filter through Whatman filter paper No. 42 and wash till it is free from chloride. Ignite and weigh as barium sulphate.

A-9.2.3 *Calculation*

$$\text{Sulphate (as SO}_4\text{), where percent by mass} = \frac{41.13\ M_1}{M_2}$$

where

M_1 = mass in g of the barium sulphate precipitate, and
M_2 = mass in g of the sample taken for the test.

A-9.3 Alternative Method

Sulphates may alternatively be determined by instrumental test method as prescribed at **A-16**.

A-10 TEST FOR IRON

A-10.1 Apparatus

A-10.1.1 *Nessler Cylinder* — 50 ml capacity.

A-10.2 Reagents

A-10.2.1 *Dilute Sulphuric Acid* — approximately 5 N.

Pour 136 ml of sulphuric acid (AR grade, 36 N) in sufficient quantity of water, mix and allow to cool. Make the volume to 1 000 ml.

A-10.2.2 *Dilute Hydrochloric Acid* — approximately 5 N.

Dilute 430 ml of hydrochloric acid (AR grade, rd 1.18) with sufficient water to make up the volume to 1 000 ml.

A-10.2.3 *Potassium Permanganate Solution* – 0.1 N.

A-10.2.4 *Ammonium Thiocyanate Solution* — approximately 7.5 N.

Dissolve 570 g of ammonium thiocyanate (AR grade) in sufficient water and make the volume to 1 000 ml.

A-10.2.5 *Amy1 Acetate* — AR or GR grade.

A-10.2.6 *Amy1 Alcohol* — AR or GR grade.

A-10.2.7 *Standard Iron Solution*

Dissolve 0.070 2 g of ferrous ammonium sulphate [$FeSO_4(NH_4)_2SO_4.6H_2O$] in water, and add 10 ml of dilute sulphuric acid (10 percent *v/v*). Dilute to exactly 100 ml in a volumetric flask. Take 25 ml of this solution into a 250 ml volumetric flask and dilute to the mark with water. One milliliter of this solution contains 0.01 mg of iron (as Fe).

A-10.3 Procedure

Dissolve 3 g of the sample in a mixture of 30 ml of water and 5 ml of dilute sulphuric acid and pass sulphur dioxide through the solution until the precipitate of iodine redissolves. Evaporate the solution to dryness on a hot plate, mix the residue with 10 ml of dilute hydrochloric acid and evaporate to dryness on a steam bath. Dissolve the residue by warming with a mixture of 3 ml of dilute hydrochloric acid and 27 ml of water. Retain 20 ml for the heavy metals test and to 10 ml apply the iron test as given in **A-10.3.1.**

A-10.3.1 Take 10 ml of solution in a 50 ml Nessler cylinder obtained in **A-10.3** add 1 drop of potassium permanganate solution and mix. Add 5 ml of ammonium thiocyanate solution and 10 ml of a mixture of equal volumes of amyl alcohol and amyl acetate. Shake vigorously and allow to separate. Any red Colour produced in the upper layer shall not be deeper than that produced in the upper layer of the standard as in **A-10.3.2.**

A-10.3.2 To 1 ml of standard iron solution in a 50 ml Nessler cylinder, add 1 ml of hydrochloric acid and dilute with water to the same volume as the acidified solution of the test sample. Add 1 drop of potassium permanganate solution and mix. Add 5 ml of ammonium thiocyanate solution and 10 ml of a mixture of equal volumes of amyl alcohol and amyl acetate. Shake vigorously and allow to separate.

A-10.4 Alternative Method

Iron may alternatively be determined by instrumental test method as prescribed at **A-17** 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.

A-11 TEST FOR HEAVY METALS

A-11.1 Apparatus

A-11.1.1 *Nessler Cylinder* — 50 ml capacity.

A-11.2 Reagents

A-11.2.1 *Dilute Acetic Acid* — approximately 5 N.

Add 285 ml of acetic acid (glacial, AR or GR grade, rd 0.88) to sufficient quantity of water and make up the volume to 1000 ml.

A-11.2.2 *Standard Lead Solution*

Dissolve 1.60 g of lead nitrate (AR or GR grade) in 50 ml of nitric acid (AR or GR grade rd 1.42), add sufficient water and make the final volume to 1 000 ml. Dilute 10 ml of this solution to 1 000 ml. One milliliter of the standard solution thus prepared contains 0.01 mg of lead (as Pb).

A-11.2.3 *Standard Iron Solution* — as in **A-10.2.7.**

A-11.3 Procedure

A-11.3.1 Take the remaining 20 ml of the solution from the iron test (**A-10**.**3**) in a 50 ml Nessler cylinder add 20 ml of water and 10 ml of dilute ammonia solution. Pass hydrogen Sulphide through the solution for a few seconds. No turbidity shall be produced. The Colour produced shall not be deeper than that of the solution prepared as in **A-11.3.2.**

A-11.3.2 Mix 2 ml of standard lead solution with 3 ml of dilute acetic acid. Add 2 ml of standard iron solution, dilute to 40 ml with water and add 10 ml of dilute ammonia solution. Pass hydrogen Sulphide through the solution for a few seconds.

A-11.4 Alternative Method

Heavy metal (as Pb) may alternatively be determined by instrumental test method as prescribed at **A-17** 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.

A-12 DETERMINATION OF SODIUM

A-12.1 Apparatus

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

A-12.2 Reagent

Standard Sodium Chloride Solution

Weigh accurately 5 g of sodium chloride and dissolve it in water. Transfer to a 1 000 ml flask and make up the volume to the mark. Dilute 10 ml of this solution to 100 ml with water immediately before use. One milliliter of the standard solution thus prepared contains 0.5 mg of sodium chloride.

A-12.3 Procedure

Dissolve 1 g of the sample in water and make up the volume to 100 ml. Divide this solution into two equal volumes. To 50 ml add 0.5 ml of standard sodium chloride solution. Spray the solution into the flame of the photometer and adjust the sensitivity to give a meter reading *S*, approaching full scale deflection, with the appropriate filter in position. Spray water into the flame and adjust the zero control to give no deflection and recheck the reading *S*. Maintaining the sensitivity, spray, the remaining 50 ml and note the meter reading *X* which should preferably be about one quarter of the full scale deflection.

A-12.4 Calculation

$$\text{Sodium (as Na), percent by mass } = \frac{X\ \times C}{M(S-X)}$$

where

C = concentration of NaCl in the standard solution, percent; and
M = mass in g of the sample used to prepare 100 ml of solution.

A-13 DETERMINATLON OF LOSS OF MASS ON DRYING AT 130 °C

A-13.1 Apparatus

A-13.1.1 *Weighing Bottle* — of approximately 100 ml capacity, 75 mm in diameter, with ground glass stopper.

A-13.1.2 *Electric Oven* — capable of maintaining a temperature of (130 ± 2) °C.

A-13.2 Procedure

Weigh 5 g of the material to the nearest 0.1 mg. Spread thin layers of the material in the weighing bottle which shall be previously dried for 30 min in the oven controlled at a temperature of 130 °C, cooled in a desiccator and weighed. Place the weighing bottle in the oven with its ground glass stopper placed slantwise. Gradually heat the oven from the starting temperature (which should not exceed 100 °C) to (130 ± 2) °C and maintain this temperature for 1 h. Remove the weighing bottle, place it in a desiccator and cool. After cooling to room temperature, weigh it again with the stopper in position.

A-13.3 Calculation

$$\text{Loss of mass on drying at 130°C, percent by mass } = \frac{(M_1 - M_2) \times 100}{M}$$

where,

M_l = mass in g of the material and weighing bottle before heating
M_2 = mass in g of the material and weighing bottle after heating, and
M = mass in g of the material taken for the test.

A-14 TEST FOR ARSENIC

A-14.1 Dissolve 1 g of the material in a mixture of 2 ml of hydrochloric acid and 1 ml of sulphuric acid and evaporate to fumes of sulphur trioxide. Add I ml of hydrochloric acid, again evaporate to fumes and dissolve the residue in 10 ml of water. Heat on a steam bath, discharge any yellow colour with hydrosulphate. Cool and dilute with water to 35 ml. This solution shall meet the requirement of arsenic test as prescribed in IS 2088.

A-14.2 Alternative Method

Arsenic may alternatively be determined by instrumental test method as prescribed at **A-17** 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.

A-15 DETERMINATION OF *p*H OF AQUEOUS SOLUTION

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

A-16 ION CHROMATOGRAPHY FOR CHLORIDES AND SULPHATES

A-16.1 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 *p*H 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.

A-16.2 Equipment

A-16.2.1 *Anion Guard Column* — A protector of the separator column.

A-16.2.2 *Anion Separator Column* — Suitable for selective separation of ions under analysis.

A-16.2.3 *Anion Suppressor Device* — Anion micro membrane suppressor is used to analyse the data.

A-16.2.4 *Detector* — Conductivity Detector.

A-16.2.5 *Software* — Software suitable for control of various operating parameters, receiving inputs and analysis of all data.

A-16.2.6 Sample loop of 100 µl, 200 µl, 500 µl or 1 000 µl be used to determine ionic concentration as per instrument manual and practice.

A-16.3 Reagents

A-16.3.1 *Glass or Polyethylene Sample Bottles.*

A-16.3.2 *Distilled Water or Deionized Water free from the Anions of interest*.

A-16.3.3 *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 litres of water.

A-16.3.4 *Micro Membrane Suppressor Solution*: (0.025 N *of sulphuric acid*) — Dilute 2.8 ml of concentrated sulphuric acid in 4 litres of water.

A-16.4 Standard Solutions

A-16.4.1 *Chloride* — Dissolve NaCl, 1.648 5 g in 1 000 ml of reagent water.

A-16.4.2 *Sulphate* — Dissolve 1.81 g of potassium sulphate in 1 000 ml of reagent water.

A-16.5 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.

A-16.6 Procedure

Dissolve between 1g to 5 g sample 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.

A-16.7 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.

A-17 DETERMINATION OF ARSENIC, IRON AND LEAD BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

A-17.1 Principle

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

A-17.2 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* A-17.2 and A-17.4)

Sl No.	Element	Wavelength (nm)	Approximately Achievable limits		Interfering Elements
			Radial viewing	Axial viewing	

			(µg)	(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)	

A-17.3 Reagents and Solutions

A-17.3.1. *Nitric Acid (65 percent) Suprapure*

A-17.3.2 *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.

A-17.3.3 *Standard Solution*

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

A-17.3.4 *Sample Preparation*

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

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

A-17.3.5 *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.

A-17.4 Instrument

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

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

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

A-17.5 Procedure

A-17.5.1 *Calibration*

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**A-17.3.5**). The relationship between concentration and intensity is linear up to six orders of magnitude. Examine the spectra of the element and make necessary adjustments (if required) for the exact peak positions and baselines to ensure proper measurements of the respective peak intensities. Flush the system with the reagent blank solution between each standard.

A-17.5.2 Before beginning the sample run, re-analyse the reference standard with the highest concentration as if it were a sample. Ensure that the concentration values do not deviate from the actual values by more than ± 5 percent (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition. Begin the sample run by flushing the system with the reagent blank solution between each sample. It is recommended to analyse a calibration check solution and the calibration blank solution every 10 samples. Analyze the sample solution and calculate the concentration in µg/ml of the lead (and/or Iron, calcium, 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.

A-17.6 Calculation

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

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

ANNEX B

(*Clause* 6)

SAMPLING OF POTASSIUM IODATE

B-1 GENERAL REQUIREMENTS OF SAMPLING

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

B-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.

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

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

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

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers in 3 single consignment of the material drawn from a single batch or manufacture shall constitute 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 ii separate lot.

B-2.2 Samples shall be tested from each lot separately for ascertaining conformity of the material to the requirements of the specification. The number of containers to be selected from a lot (*n*) 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

SI No. (1)	Lot Size *N* (2)	Number of Containers to be Selected *N* (3)
i)	4 to 50	3
ii)	51 to 100	4
iii)	101 to 150	5
iv)	151 to 300	7
v)	301 and above	10

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.

B-2.3 These containers shall be selected at random from the lot. For random selection procedure, guidelines can be had from IS 4905. In case IS 4905 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/r*. Every r^{th} container thus counted shall be taken out

B-3 PREPARATION OF TEST SAMPLES AND REFEREE SAMPLE

B-3.1 For drawing samples, an auger shall be used. Introduce the auger in different parts of each container to draw an appropriate quantity of the sample from each container.

B-3.2 Out of the portions collected from all the selected containers, an equal quantity of the material shall be taken out and mixed together to form a composite sample of about 250 g. The composite sample so formed shall be divided into three equal parts, one for the purchaser, and another for the supplier and the third to be used as a referee sample.

B-3.3 The remaining portion of the sample 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.

B-3.4 All the individual and the composite samples shall be immediately transferred to separate glass bottles which shall be labelled with full identification particulars of the samples.

B-3.5 The referee samples 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.

B-4 NUMBER OF TESTS

Tests for potassium iodate content, matter insoluble in water and loss on drying shall be conducted on each of the individual samples and tests for all other requirements given in **4.1** and Table **1** shall be conducted on the composite sample.

B-5 CRITERIA FOR CONFORMITY

A lot shall be considered as conforming to the requirements of this specification if the stipulations laid down in **B-5.1** and **B-5.2** are fulfilled.

B-5.1 for Potassium Iodate

From the individual test results for potassium iodate content the mean ($\overline{X}$) and the range (R) or mean range ($\overline{\mathrm{R}}$) shall be calculated. (Range is the difference between the maximum and the minimum of the test results). When the sample size is 10, the test results shall be arranged in two groups of 5 each in the order of their occurrence and the range for each group computed. R will be the mean of these two R*'s*. The lot shall be deemed satisfactory for this characteristic if ($\overline{X} – 0.6\ R$) or ($\overline{X} – 0.6\ \overline{R}$) is greater than or equal to the minimum requirement specified in Table 1.

B-5.2 For Other Characteristics

The test results for all other characteristics prescribed in **4.1** and Table 1 shall satisfy the relevant requirements.