भारतीय मानक
Indian Standard

IS 17439 : 2020

पॉलीफॉस्फोरिक अम्ल — विशिष्टि

Polyphosphoric Acid — Specification

ICS 71.060

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

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FOREWORD

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

Polyphosphoric acids are a series of oxyacids of phosphorus with the general formula $H_{n+2}P_nO_{3n+1}$ formed by condensation of orthophosphoric acid molecule and containing a backbone chain consisting of alternating P and O atoms bonded together. Polyphosphoric acid is commercially produced either by dehydration of H_3PO_4 at high temperature (known as wet process) or by heating P_2O_5 dispersed in H_3PO_4 (known as dry process). The dehydration method tends to produce short chain whereas the dispersion method usually produces chains with more than 10 repeated units. Polyphosphoric acid is non-oxidizing agent having powerful dehydration properties with moderate acidity. Polyphosphoric acid is used to catalyse cyclization of acids, esters, ketones, aldehydes, acetals, alcohols and alkanes to aromatize ring derivatives. The first member of the series is pyrophosphoric acid (n=2) and the series includes the highly polymeric metaphosphoric acid. The higher acids generally exist in an equilibrium mixture. PPA is used in synthetic processes. PPA is widely used in industries including medicine, aromatics, leather and chemical industry. It is used as a catalyst for dimethyl carbonate synthesis from urea and methanol. PPA acts like an adsorbent in ammonia generated process. In the form of silicon dioxide-polyphosphoric acid, it forms a reusable,easy to handle heterogenous catalyst. PPA can also be used as a substitute for orthophosphoric acid.

The list of experts who had made significant contribution to the formulation of this standard is given at Annex C.

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 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

POLYPHOSPHORIC ACID — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for polyphosphoric acid.

2 REFERENCES

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

IS No.	*Title*
265 : 1993	Hydrochloric acid — Specification (*fourth revision*)
266 : 1993	Sulphuric acid — Specification *(third revision)*
323 : 2009	Rectified spirit for industrial use — Specification (*second revision*)
1060 (Part 1) : 1966	Methods of sampling and test for paper and allied products: Part 1 (*revised)*
1070 : 1992	Reagent grade water (*third revision*)
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 35) : 1988	Silica *(first revision)*
(Part 48) : 1994	Mercury *(first revision)*
(Part 55) : 2003	Aluminium *(first revision)*

3 GRADE

The material shall be of the following two grades:

a) Technical grade (content 95 percent, 105 percent on H_3PO_4 basis)

b) Reagent grade (content 115 percent on H_3PO_4 basis)

4 REQUIREMENTS

4.1 The material shall be in the form of odourless, colourless highly viscous liquid. It should be diliquescent and soluble in water.

NOTE — At lower temperature, PPA appears as glass and becomes liquid/fluid, when heated to 50-60 °C.

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

5 PACKING AND MARKING

5.1 Packing

The material shall he packed in containers as agreed to between the purchaser and the supplier.

When the material is supplied in screw stoppered stone bottles or glass carbuoys, the container shall be fitted with leak-tight stopper and, where necessary, provided with asbestos or rubber washers. The stoppers shall further be sealed by a putty made of china-clay, or a mixture of sodium silicate and asbestos flour.

The bottles and jars shall be packed in suitable pent top packing cases. These shall be placed in an upright position on a layer of sand, or coal ash, or dry earth, and the empty surrounding space in the packing case shall also be filled with the same material to prevent movement. Carbuoys shall be packed in suitable iron hampers or wooden crates stuffed with chemically inert materials, like clay or fullers earth.

5.2 Marking

5.2.1 The containers shall be suitably marked with the name and grade of the material; manufacturer's name and recognized trademark, if any; date of manufacture and net mass. They shall also display prominently the words, in red letters not less than 2 cm high, `CORROSIVE', HANDLE WITH CARE'.

The containers shall be labelled as shown in Fig. 15 of IS 1260 (Part 1).

Table 1 Requirements for Polyphosphoric Acid

(*Clause* 4.2)

Sl No.	Characteristic	Technical Grade 95 Percent 105 Percent		Reagent Grade (115 Percent)	Annex
(1)	(2)	(3)		(4)	(5)
i)	Relative density, g/ml (27°C)	1.8±0.01	1.94 ±0.01	2.06±0.01	A-2
ii)	H_3PO_4 (percent by mass, *Min)*	95.0	105.0	115.0	A-3
iii)	P_2O_5 (percent by mass, *Min)*	68.82	76.07	83.32	A-3.4
iv)	Fe (ppm, *Max*)	20	20	20	A-4 and A-18
v)	Cl (ppm, *Max*)	5	5	5	A-5 and A-17
vii)	SO_4^{2-} (ppm, *Max*)	50	50	50	A-6 and A-17
viii)	As (ppm, *Max*)	1	1	1	A-7 and A-18
ix)	Pb (ppm, *Max*)	1	1	1	A-8 and A-18
x)	Nitrates (ppm, *Max*)	5	5	5	A-9
xi)	Antimony (ppm, *Max*)	15	15	-	A-10
xii)	Manganese (ppm, *Max*)	-	-	0.4	A-11 and A-18
xiii)	Molybedenum (ppm, *Max*)	10	10	-	A-12 and A-18
xiv)	Mercury (ppm, *Max*)	0.3	0.3	-	A-13 and A-18
xv)	Aluminium (ppm, *Max*)	10	10	-	A-14 and A-18
xvi)	Calcium and Magnesium, ppm (*Max*)	-	-	6	A-15 and A-18
xvii)	Silica as SiO_2 (ppm, *Max)*	-	-	50	A-16

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

Representative samples of the material shall be drawn as prescribed in Annex B.

ANNEX A

(*Clause* 5.2, *Table* 1)

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 DETERMINATION OF RELATIVE DENSITY

A-2.1 General

This shall be determined by a capillary-stoppered relative density bottle.

A-2.2 Procedure

Clean the relative density bottle first with a saturated solution of chromic acid in concentrated sulphuric acid followed by repeatedly rinsing with distilled water to remove traces of chromic acid and then with alcohol. Dry the bottle, breathe on it to dissipate any static charges that may have been formed (a precaution that shall be taken also before every subsequent weighing), bring it to room temperature and weigh. Fill the weighed bottle with distilled water and place it in the constant temperature bath maintained at 27.0 ± 0.5°C for 90 min to attain temperature equilibrium. Wipe excess liquid from the top of the stopper, remove the bottle from the bath, wipe the outside to dry, weigh the bottle along with its content after 15 minutes conditioning at room temperature (maintained between 25-27°C). The difference between the masses of the filled and empty bottle gives the water equivalent, that is, the mass in air of the water contained in the bottle at 27°C. Empty the bottle, rinse several times with alcohol and finally with ether. Remove the ether fumes with the aid of an air blast and permit the bottle to dry thoroughly. Fill the bottle with the material, warming both the material and bottle to assist filling and removal of air bubbles, and then bring the bottle and contents to 27.0 ± 0.5°C. When the temperature is constant, insert the capillary stopper which has also been brought to 27.0 ± 0.5°C and remove excess liquid from the top of the stopper. Remove the bottle from the bath, wipe the outside dry, weigh the bottle along with its content after 15 minutes conditioning at room temperature (maintained between 25-27°C).

A-2.3 Calculation

To obtain the relative density of the material at 27°C, divide the net mass of polyphosphoric acid by the water equivalent of the bottle.

A-3 DETERMINATION OF PHOSPHORIC ACID CONTENT

A-3.1 General

Polyphosphoric acid is first hydrolysed to orthophosphoric acid which may be determined by either of the three methods, namely, Method A, Method B and Method C. In case of dispute, Method A shall be adopted.

A-3.1.1 *Outline of the Method*

The material is titrated against standard solution of alkali.

A-3.2 Method A

A-3.2.1 *Reagents*

A-3.2.1.1 *Standard sodium hydroxide solution — 1 N.*

A-3.2.1.2 *Bromocresol green indicator* — Dissolve 0.1g of bromocresol green in 100 ml of rectified spirit (*see* IS 323).

A-3.2.1.3 *Sodium dihydrogen phosphate solution* — Approximately 5·5 percent solution (*m*/*v*).

A-3.2.2 *Procedure*

Weigh accurately about 4 g of the material in a conical flask and dilute with 60 ml of water. Titrate against standard sodium hydroxide solution to *p*H 4·5 using bromocresol green indicator. The end point may be judged by comparing the colour with 60 ml of sodium dihydrogen phosphate solution containing the same amount of indicator as used in the solution being titrated.

A-3.2.3 *Calculation*

Phosphoric acid (as H_3PO_4), percent by mass

$$= \frac{9.80 \times V \times N}{M}$$

where

V = volume in ml, of standard sodium hydroxide solutionused;

N = normality of standard sodium hydroxide solution; and

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

A-3.3 Method B

A-3.3.1 *Reagents*

A-3.3.1.1 *Mixed screened indicator* — Prepare by mixing together one part by volume of methyl orange solution (0·1 percent, aqueous), one part by volume of xylene cyanol FF solution (0·1 percent, aqueous) and three parts by volume of phenolphthalein solution (0.1 percent, alcoholic).

NOTES

1 When stored in brown bottles, the indicator has shelf life of about two months. If, at any stage, the indicator shows precipitation or a tendency of separation of constituents, it should be discarded.

2 The indicator, when used in aqueous solution (8 to 10 drops per 100 ml of solution), has a pink violet colouration at *p*H below 4. On raising the *p*H, the solution becomes successively colourless, faintly green and finally yellowish green at *p*H of about 4·5. The colour and intensity remain constant on further raising the *p*H value till about *p*H 8.8. At this stage, it becomes nearly colourless again. When pH is raised to 9·0, faint pink colour develops again. The colour is intensified on further increase in *p*H value.

A-3.3.1.2 *Standard sodium hydroxide solution* — 1N.

A-3.3.1.3 *Sodium chloride*

A-3.3.2 *Procedure*

Weigh accurately about 4 g of the material in a conical flask, dilute with 60 ml of freshly boiled water and add 8 to 10 drops of mixed screened indicator. Titrate against standard sodium hydroxide solution. When the colour of the solution becomes yellowish green, add a few more millilitres of alkali (the colour of the solution stays yellowish green), and add 10 g of sodium chloride. Continue the titration till the appearance of the first faint permanent pink colour. Note the total volume of the alkali added.

A-3.3.3 *Calculation*

Polyphosphoric acid (as H_3PO_4), percent by mass

$$= \frac{4.90 \times N \times V}{M}$$

where

N = normality of standard sodium hydroxide solution;

V = volume in ml, of standard sodium hydroxide solution used; and

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

A-3.4 Method C-Determination of P_2O_5 (Spectrophotometric method)

A-3.4.1*General*

Polyphosphoric acid is first hydrolysed to orthophosphoric acid which may be treated with an acidified molybdovanadate reagent to form the yellow coloured molybdovanado phosphoric acid complex. $(NH_4)_3\ PO_4.NH_4\ VO_3.\ 16MOO_3$ with $NH_4\ VO_3$, and $(NH_4)_6\ Mo_7O_{24}.4H_20$.

The reaction is sensitive to variations in acidity, and care must be taken to hold the acidity within limits. As the heat intensifies the colour, sample, and the standard must be at same temperature when the absorbance is measured.

A-3.4.2 *Reagents*

A-3.4.2.1 *Reagent A*

a) Dissolve 1.12 g of ammonium meta vanadate into water and then add nitric acid 250 ml (solution A).

b) Dissolve 27 g of ammonium molybdate in water. If the solution is turbid add little NH_3 solution till the solution is clear (solution B)

Mix solution B with A and dilute to 1 litre with water (mark it as Reagent A). Store the reagent-A in ambered coloured bottle.

A-3.4.2.2 *Standard P_2O_5 solution*

Dissolve 19.174 g of potassium dihydrogen phosphate (KH_2PO_4) in 1 litre volumetric flask with water. Add 2-3 ml of HNO_3 in order to preserve the solution. Dilute to the mark and mix well. One millilitre of this solution contains 10 mg of P_2O_5.

A-3.4.2.3 *Standard solutions of 3,4,5 and of 6 mg of P_2O_5 per 10 ml of solution*

Transfer 30, 40 ml, 50 and 60 ml of standard P_2O_5 solution by means of burette into separate 1-litre capacity volumetric flasks and dilute to the mark with water upto mark. The resultant solutions contains 3,4,5 and 6mg of P_2O_5 per 10 ml respectively.

A-3.4.3 *Procedure*

A-3.4.3.1 Weigh accurately about 4 g (M) of material in a 200 ml beaker, dissolve in 50 ml water and quantitatively transfer to a 500ml volumetric flask by rinsing with water. Make up volume up to mark. Mark it as Solution-T. Transfer 10 ml of solution –T into a 100 ml volumetric flask and dilute it to the mark with water. From this flask transfer 10 ml of the solution by pipette into another 100 ml volumetric flask, dilute with 30 ml water, add 20 ml of reagent-A (*see* **A-3.4.2.4.1**), make up the volume, mix well and allow to stand for half an hour.

A-3.4.3.2 Similarly transfer 10 ml aliquots of standard solutions (*see* **A-3.4.2.3**) containing 3.0, 4.0, 5.0 mg and 6.0 mg of P_2O_5 to 100 ml volumetric flasks, dilute with 30 ml water, add 20 ml of reagent –A (*see* **A-3.4.2.1**), make up the volume, shake well and allow to stand for half an hour.

Prepare reagent blank by diluting 20 ml of the reagent-A to 100 ml.

A-3.4.3.3 Adjust the spectrophotometer to read 100 percent transmittance for reagent blank using 10mm cell at 420 nm wave length. Then measure absorbance

for different concentration of standard for example, 3.0, 4.0, 5.0 and 6.0 mg of P_2O_5 and plot a graph of absorbance against different concentration of P_2O_5 (in mg). Similarly measure the absorbance for the sample and from the graph determine the mg P_2O_5 in the sample solution (P) and calculate the mg of P_2O_5 in the sample by calculation at **A-3.4.4**.

A-3.4.4 *Calculation*

P_2O_5, percent by mass in test sample, $Z = \frac{(50 \times P)}{M}$

Polyphosphoric acid (as H_3PO_4), percent by mass = percent $P_2O_5 \times 1.3803 = Z \times 1.3803$

A-4 TEST FOR IRON

A-4.1 General

Trivalent iron is reduced by means of hydroxyl-ammonium chloride and a bivalent iron-2, 2'-bipyridyl complex at *p*H 3.1, at a temperature of 75°C is formed. Photometric measurement of the coloured complex is done at a wavelength of about 520 nm.

A-4.2 Apparatus

A-4.2.1 *p*H *Meter — Fitted* with glass electrode.

A-4.2.2 *Spectrophotometer* — Provided with a 1 cm cell.

A-4.3 Reagents

A-4.3.1 *Concentrated Hydrochloric Acid — See* IS 265.

A-4.3.2 *2,2'-Bipyridyl Solution* — Dissolve 0.50 g of 2,2'-bipyridyl in 10 ml of concentrated hydrochloric acid and dilute to 100 ml with water.

A-4.3.3 *Ammonium Acetate Solution* — Dissolve 300 g of ammonium acetate in water and make the volume to 1000 ml.

A-4.3.4 *Hydroxylammonium Chloride* — Dissolve 10 g of hydroxyl-ammonium chloride ($NH_2OH.HC1$) in water and dilute to 100 ml.

A-4.3.5 *Standard Iron Solution* — Dissolve 0·702 g of ferrous ammonium sulphate [Fe $(NH_4)_2(SO_4)_2.6H_2O$] in 20 ml of 10 percent sulphuric acid and dilute with water to exactly 500 ml. Further dilute 100 ml of this solution to 1 000 ml. One millilitre of the diluted solution is equivalent to 0.02 mg of iron (as Fe).

A-4.4 Procedure

A-4.4.1 *Preparation of Sample Solution*

Weigh 50 g of the material to the nearest 0·01 g in a small glass beaker. Transfer quantitatively to a 100 m1 volumetric flask and make up to the mark with carbon dioxide-free water. Mix and use the sample solution for tests.

A-4.4.2 *Blank Test*

At the same time as the analysis, carry out a blank test using the procedure described in **A-4.4.1.4** and the same quantities of all reagents.

A-4.4.3 *Preparation of Calibration Curve*

Proceed the method as prescribed below:

a) *Preliminary check of p*H — Place 5 ml of concentrated hydrochloric acid in a beaker of suitable capacity (100 ml for example), dilute to approximately 50 ml, add 1 ml of hydroxylammonium chloride solution and 5 ml of 2,2'-bipyridyl solution. Allow to stand for about ten minutes and using the *p*H meter, adjust the *p*H of the solution to 3.1 by addition of ammonium acetate solution. Note the quantity of ammonium acetate added for the *p*H adjustment and then discard the solution.

b) *Preparation of standard solutions* — Into each of a series of five beakers of suitable capacity (100 ml, for example), place the quantities of standard iron solution indicated below:

Standard Iron Solution (ml)	Corresponding to Iron (Fe) (μg)
*0	0
5.0	100 (5.0 × 0.02mg = 0.1mg)
10.0	200 (10.0 × 0.02mg = 0.2mg)
15.0	300 (15.0 × 0.02mg = 0.3mg)
25.0	500 (25.0 × 0.02mg = 0.5mg)

*Compensation solution

Then add 5 ml of concentrated hydrochloric acid and dilute to approximately 50 ml.

c) *Colour development* — To each of the above-mentioned solutions, add 1 ml of hydroxylammonium chloride solution and 5 ml of 2,2'-bipyridyl solution. Allow to stand for about ten minutes, then add the quantity of ammonium acetate solution determined following the procedure of **A-4.4.1.3**(a) for adjusting *p*H of 3.1. Heat the solutions on a water-bath at approximately 75°C for about fifteen minutes and then cool to room temperature. Transfer to 100 ml one-mark volumetric flasks, dilute to the mark and mix thoroughly.

d) *Photometric measurement* — Measure the optical density of each solution using the spectrophotometer at a wavelength of about 520 nm, adjusting the instrument to zero optical density using as reference the compensation solution.

e) *Preparation of calibration graph* — Prepare a calibration graph for iron (Fe) content in micrograms per 100 ml of the standard solution as abscissa and the corresponding values of optical density as ordinate.

A-4.4.4 *Determination*

Pipette out 10 ml of the diluted sample solution (*see* **A-4.4.1.1)** into a beaker of suitable capacity (100 ml) and dilute to approximately 50 ml. Add 1 ml of hydroxylammonium chloride solution and 5 ml of 2,2'-bipyridyl solution. Allow to stand for about ten minutes, then using the *p*H meter, adjust the *p*H of the solution to 3.1 by addition of ammonium acetate solution. Heat the solution on a water-bath to approximately 75°C for about fifteen minutes and cool to room temperature. Transfer the solution quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark and mix thoroughly. Measure the optical density of the solution adjusting the instrument to zero optical density with the blank test solution and following the procedure described in **A-4.4.1.3**(d).

A-4.4.5 *Calculation*

By reference to the calibration graph, read the iron content corresponding to the photometric measurement of the sample and calculate.as given below:

$$\text{Iron (as Fe), ppm} = \frac{A \times 100 \times 100}{E}$$

where

A = mass in mg, of iron determined in the sample solution, and

E = mass in g, of the sample taken for the test in **A-4.4.1.1.**

A-4.5 Alternative Method

Iron may alternatively be estimated by instrumental method as **A-18.**

A-5 TEST FOR CHLORIDES

A-5.1 General

Opalescence produced by the material with silver nitrate is compared with that of a control with known chloride content.

A-5.2 Apparatus

A-5.2.1 *Nessler Cylinder* — 50 m1 capacity.

A-5.3 Reagents

A-5.3.1 *Dilute Nitric Acid* — Approximately 4 N, free from chlorides.

A-5.3.2 *Silver Nitrate Solution* — Approximately 5 percent. Prepared by dissolving 5g $AgNO_3$ (GR) in 100 ml water containing 2 ml nitric acid or by dissolving proportionate amount of $AgNO_3$ in reduced volume of water. If any turbidity observed, filter through sintered glass crucible (5) under section and preserved the solution in ambered coloured glass bottle.

A-5.3.3 *Standard Chloride Solution* — Dissolve 0.164 g of sodium chloride in water and make up the volume to 1 000 ml. Further dilute 10 ml of this solution to 100 ml. One millilitre *of this* solution contains 0·01 mg of chloride (as Cl).

A-5.4 Procedure

Transfer 4 ml of diluted sample solution **(A-4.4.1.1)** to a Nessler cylinder. Add 1 ml of dilute nitric acid, 1 ml of silver nitrate solution and make up the volume to 50 ml. Carry out a control test in another Nessler cylinder using 1 ml of standard chloride solution and the same quantities of other reagents. Stir the solutions with a glass rod and set aside for five minutes. Compare the opalescence produced in the two cylinders.

A-5.5 The material shall be taken to have not exceeded the limit prescribed if the opalescence produced in the test with material is not greater than that produced in the control test.

A-5.6 Alternate Method

Chlorides may alternatively be estimated by the Instrumental method as prescribed at **A-17.**

A-6 TEST FOR SULPHATES

A-6.1 General

The sulphate limit shall be estimated by comparison of the turbidity produced by barium chloride with that of a control solution.

A-6.2 Method-A

A-6.2.1 *Apparatus*

A-6.2.1.1 *Nessler cylinder* — 50-ml capacity.

A-6.2.2 *Reagents*

A-6.2.2.1 *Dilute hydrochloric acid* — Approximately 5 N.

A-6.2.2.2 *Barium chloride solution* — Approximately 10 percent (*m*/*v*). Prepared by dissolving 10 g $BaCl_2$ (GR) in 100 ml water or by dissolving proportionate amount of $BaCl_2$ in reduced volume of water. If any turbidity observed, filter through sintered glass crucible (4) under section and preserved the solution in glass bottle.

A-6.2.2.3 *Standard sulphate solution* — Dissolve 0·124 g of ignited sodium sulphate in water and dilute to 1 000 ml. One millilitre of this solution contains 0·01 mg of sulphate (SO_4).

A-6.2.3 *Procedure* — Take 1·0 g of the material in a Nessler cylinder, and add 2 ml of dilute hydrochloric acid and 5 ml of barium chloride solution. Make the

volume to 50 ml. Carry out a control test in another Nessler cylinder using 5 ml of standard sulphate solution and the same quantities of the other reagents. Compare the turbidity produced in the cylinders.

A-6.2.4 The limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced by the material is not greater than that produced in the control test.

A-6.3 Method-B

A-6.3.1 Sulphate is determined by spectrophotometer, developing the turbidity of barium sulphate. The turbidity of a dilute barium sulphate suspension is difficult to reproduce. It is, therefore, essential to adhere rigidly to the conditions of experimental procedure.

A-6.3.2 *Reagents*

A-6.3.2.1 *Standard sulphate solution*

1ml equivalent to 1 mg H_2SO_4 (weighed quantity 1.0204 g of concentrated H_2SO_4 (98 percent) transfer into a beaker carefully having small quantity of water. Transfer this to a 1 000 ml volumetric flask and makeup the volume with water). Transfer 50 ml of this stock solution to a 250 ml volumetric flask and dilute to the mark. 5 ml of this solution is equivalent to 1 mg H_2SO_4.

A-6.3.2.2 *Conditioning reagent*

Mix 50 ml of glycerine with a solution containing 30 ml of concentrated hydrochloric acid, 300 ml of water and 75 g of sodium chloride.

A-6.3.2.3 *Barium chloride*

Use crystals of barium chloride that pass through 850-micron IS Sieve and retain on 500-micron IS Sieve.

A-6.3.3 *Calibration Curve*

Transfer 5, 10, 15, 20, 25 and 30 ml of the standard sulphate solution (*see* **A-6.3.2.1**) from a calibrated burette into separate 100 m1 volumetric flasks. To each flask add 5 ml of the conditioning reagent and dilute to 100 ml with water. Mix well. Add 0.3 g of solid barium chloride to each flask, and shake for 1 min by inverting each flask once per second, until barium chloride dissolve completely. Allow to stand for 4 min. Prepare blank for reagents with distilled water and equal quantity of all reagents except standard solution. Measure the percent transmission (T) for each standard solution on spectrophotometer at 420 nm wavelength after adjusting the light transmission for blank at 100 percent. Obtain log T and plot a calibration graph for concentration of H_2SO_4 (in mg) versus log T, passing through origin.

A-6.3.4 *Procedure*

Transfer 10 ml of dilute sample solution **(A-4.4.1.1)** to a 100 ml volumetric flask. Add 5 ml of the conditioning reagent and dilute to the mark with water. Mix well. Add 0.3 g of barium chloride crystals and shake for 1 min by inverting the flask once per second. Allow to stand for four minutes. Prepare a reagent blank. Measure percent transmission (T) on spectrophotometer at 420 nm wavelength by adjusting light transmission for blank at 100 percent. From the log T, evaluate the concentration of H_2SO_4 (in mg) in solution, from the calibration graph.

A-6.3.5 *Calculation*

$$\text{Sulphate, ppm} = \frac{M_1 \times F \times 0.9795}{M}$$

where

M_1 = mass in mg, of H_2SO_4 obtained from the calibration graph;

F = dilution factor; and

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

A-6.4 Alternate Method

Sulphates may alternatively be estimated by the Instrumental method as prescribed at **A-17.**

A-7 TEST FOR ARSENIC

A-7.1 Principle of Test

Arsenic present in sample is converted into AsH_3 and the stain produced by arsine on mercuric bromide paper is compared with a standard stain.

A-7.2 Reagents

A-7.2.1 *Concentrated Hydrochloric Acid* **(***see* IS 265).

A-7.2.2 *Hydrazine Sulphate*

A-7.2.3 *Sodium Bromide*

A-7.3 For Technical Grade

A-7.3.1 *Preparation of Test Solution*

Weigh 1.0 g of the material and transfer to a small distillation flask. Add 10 ml of water, 15 ml of concentrated hydrochloric acid, 0.25 g of hydrazine sulphate and 0.25 g of sodium bromide. Connect the flask to a condenser and distil the contents till 20 ml of the distillate are collected. Use this distillate for carrying out the test for arsenic, as described in IS 2088, using for comparison of a stain obtained with 0.001 5 mg of arsenic trioxide. Reserve the residue in the distillation flask.

A-7.4 For Reagent Grade

Weigh 1.0 g of the material and carry out the test for arsenic by the modified Gutzeit method as prescribed in **5.1** or spectrophotometric method as prescribed in **5.2** of IS 2088, for comparison as per **5.1**, use a stain obtained with standard of 0.0015 mg of arsenic trioxide.

In case of dispute, the spectrophotometric method shall be the referee method.

A-7.5 Alternative Method

Arsenic may alternatively be estimated by instrumental method as **A-18.**

A-8 TEST FOR LEAD (AS PB)

A-8.1 Method A

AA-8.1.1 *General*

The colour developed by the material with sodium sulphide solution is compared with that of a control solution.

A-8.1.2 *Apparatus*

8.1.2.1 *Nessler cylinder* — 50 m1 capacity.

8.1.3 *Reagents*

A-A-8.1.3.1 *Ammonium hydroxide* — 5 N.

A-8.1.3.2 *Dilute acetic acid-1:1*

A-8.1.3.3 *Sodium sulphide solution* — 12 percent *(m/v)*

A-8.1.3.4 *Standard Lead solution*

Dissolve 0·160 g of lead nitrate in 90 ml of water to which 1 ml of nitric acid has been added, and dilute to 100 ml. Keep this solution in lead-free stoppered bottle. Further dilute 10 ml of this solution with water to 1 000 ml. One millilitre of this solution contains 0.01 mg of lead (as Pb).

A-8.1.4 *Procedure*

Dilute 10 g of the material with 10 ml of water in a 150 ml beaker, neutralize with ammonium hydroxide solution, add sufficient dilute acetic acid to render the solution acidic. Transfer this solution to a 50 ml Nessler cylinder and dilute with water to mark and add 0.5 to 1ml of sodium sulphide solution. Carry out a control test using 1 ml of standard lead solution, 5ml of dilute acetic acid in a 50 ml Nessler cylinder diluting to mark with water and adding 0.5 to 1ml of sodium sulphide solution.

A-8.1.4.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

A.8.2 Method B (Spectroscopic Method)

A-8.2.1 *Principle*

A sample containing microgram quantities of lead (4 g) is extracted with dithizone solution in chloroform. The extraction is carried out in the presence of strong ammoniacal citrate-cyanide reducing agent *(p*H 10 to 11.5). The quantity of lead present in the sample is determined spectrophotometrically by measuring the absorbance at 510 nm in chloroform extract containing the lead dithizonate complex.

A-8.2.2 *Minimum Detection Limit*

1.0 µg Pb/10 ml dithizone solution (extract).

A-8.2.3 *Apparatus*

A-8.2.3.1 *Spectrophotometer,* for use at 510 nm with a path length of 1 cm or longer.

A-8.2.3.2 *pH meter*

A-8.2.3.3 *Standard volumetric glasswares*

A.8.2.3.4 *TEF beaker,* 100 ml for acid digestion.

A-8.2.3.5 *Separatory funnels,* 250 ml and 500 ml.

All glasswares are to be cleaned with 1:1 HNO_3, and rinsed thoroughly with distilled water.

A-8.2.4 *Reagents*

A-8.2.4.1 *Quality of reagents*

Only analytical or equivalent grade reagents, unless specified otherwise, are to be used. All reagents are to be prepared in lead-free distilled water.

A-8.2.4.2 *Stock lead solution*

Dissolve 0.159 9 g lead nitrate [$(Pb(NO_3)_2$, minimum purity, 99.5 percent (*w/w*)] in about 200 ml of water. Add 10 ml concentrated HNO_3 and dilute to 1 000 ml with water, 1.0 ml of this solution will contain 100 µg of Pb.

A-8.2.4.3 *Standard lead solution*

Dilute 2.0 ml of stock lead solution to 100 ml with water, 1.0 ml of this solution will contain 2 µg of Pb.

Nitric Acid — Concentrated (18N).

Nitric Acid — Dilute —20 percent, *v/v*.

Ammonium Hydroxide — Concentrated (14N).

Ammonium Hydroxide — Dilute 10 percent, *v/v* and 1 percent, *v/v.*

Citrate-Cyanide Reducing Solution — Dissolve 200 g anhydrous ammonium citrate [$(NH_4)_2$ $HC_6H_5O_7$] 10 g anhydrous sodium sulphite (Na_2SO_3), 5 g hydroxylamine hydrochloride, 20 g potassium cyanide (KCN) in water and dilute to 500 ml, and mix with one litre of concentrated NH_4OH.

CAUTION — KCN is a poisonous solution. Handle with extreme care and do not pipette by mouth.

A-8.2.4.4 *Stock dithizone solution*

Dissolve 25 mg dithizone in about 50 ml chloroform ($CHCl_3$) taken in a 200 ml beaker and filter through

Whatman No. 42 (or equivalent) filter paper. Collect the filtrate and two washings (10 ml each) in a 250 ml conical flask. Transfer the combined filtrate to a 500 ml separatory funnel. Add about 100 ml 1 percent (*v/v*) NH_4OH solution, shake moderately for about 1 min. Transfer the $CHCl_3$ layer to another 250 ml separatory funnel retaining the orange-red aqueous layer in the 500 ml separatory funnel. Repeat the extraction (of the $CHCl_3$ layer) with 100 ml of 1 percent (*v/v*) NH_4OH solution, transfer the $CHCl_3$ layer to another 250 ml separatory funnel and the aqueous layer to the original 500 ml separatory funnel containing the first extract. One more repetition, of extraction and transferring to the main aqueous layer is carried out. To the combined aqueous extract in the 500 ml separatory funnel add 1:1 HC1 in 2 ml portions, mixing after each addition, until dithizone precipitation is complete and the solution is no longer orange-red. Extract the precipitated dithizone with three 25 ml portions of $CHCl_3$. Dilute the combined extract to 250 ml with $CHCl_3$, 1 ml of this solution will contain 100 µg of dithizone.

A-8.2.4.5 *Working dithizone solution*

Dilute 100 ml stock dithizone solution to 250 ml in a standard volumetric flask with $CHCl_3$, 1 ml of this solution will contain 40 µg of dithizone.

A-8.2.5 *Procedure*

A.8.2.5.1 *Sample digestion*

Digest all samples for lead as per standard digestion procedure using minimum amount of HNO_3-H_2SO_4 and HNO_3-$HClO_4$. Dilute the acidified sample (*p*H = 2) to 100 ml and add 20 ml of dilute (20 percent, *v/v*) HNO_3, filter if required through a filter paper (Whatman No. 41 or equivalent), and transfer it to a 250 ml separatory funnel. Add 60 ml ammoniacal citrate-cyanide solution, mix and cool to room temperature. Add 10 ml of dithizone working solution. Shake the stoppered funnel vigorously for about 30s, allow to stand (to get two separate layers). Discard 1-2 ml $CHCl_3$ layer and then fill the absorption cell. Measure the absorbance at 510 nm using working dithizone solution as reagent blank.

A-8.2.5.2 *Calibration curve*

Plot a calibration curve using at least five standard lead solutions, after adding 50 ml ammoniacal citrate-cyanide solution to the individual lead standard solutions and extracting the same with 10 ml of dithizone working solution.

A-8.2.6 *Calculation*

mg Pb/litre=µg (in 10 ml extract obtained from calibration curve)/Volume of sample (ml)

A-8.3 Alternative Method

Lead may alternatively be estimated by instrumental method as **A-18**.

A-9 TEST FOR NITRATES

A-9.1 Procedure

Dilute 3.48 g of the sample in 10 ml of water and add 5 mg of sodium chloride, 0.1 ml of indigo carmine solution, and add 10 ml of sulphuric acid. The blue colour shall not disappear entirely within 5 min.

A-10 TEST FOR ANTIMONY

A-10.1 General

The stain produced by stibine on mercuric bromide paper is toned with auric chloride and the toned stain compared with standard stain.

A-10.2 Reagents

In addition to the reagents given in **A-7.2,** the following reagents are also required.

A-10.2.1 *Dilute Ammonium Hydroxide* — 1 : 3 (*v/v*).

A-10.2.2 *Chloro Auric Acid Solution* — Approximately 1 percent solution of $HAuCl_4.4H_2O$.

A-10.2.3 *Standard Antimony Solution* — Dissolve 2·291 g of potassium antimonyl tartrate in water and dilute to 1 litre. Pipette out 10 ml of this solution and further dilute with water to 1 litre. Dilute 100 ml of the diluted solution again to 1 litre. One millilitre of the final solution contains 0·001 mg of antimony (as Sb_2O_3).

A-10.3 Procedure

Take the residue from the distillation flask preserved in **A-7.3.1** and carry out the test as for arsenic **(A-7.3.1).** Develop the produced stain by dipping the paper in dilute ammonium hydroxide for three minutes, followed by washing five times with water. Then tone the paper by dipping in chloro auric acid solution for five minutes. Finally wash the paper five times with water. Compare the violet stain produced with a standard stain obtained following the similar procedure, using 18 ml of standard antimony solution.

A-10.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the length and intensity of colour of the stain with the material are not greater than those of the standard stain.

A-11 TEST FOR MANGANESE

A-11.1 Apparatus

A-11.1.1 *Nessler Cylinders* — 100-m1 capacity.

A-11.2 Reagents

A-11.2.1 *Phosphoric Acid, 85 Percent* (manganese free)

A-11.2.2 *Potassium Periodate*

A-11.2.3 *Dilute Sulphuric Acid, 50 Percent (v/v)*

A-11.2.4 *Standard Manganese Solution A* — Dissolve 0·406 g of manganous sulphate tetrahydrate or 0·308 g of manganous sulphate monohydrate in a mixture of 50 ml of dilute sulphuric acid and 100 ml of water, and make up the volume to 1 000 ml with water. One millilitre of this solution contains 0·1 mg of manganese (as Mn).

A-11.2.5 *Standard Manganese Solution B* — Take 100 ml of standard manganese solution A (**A-11.2.4**) and dilute it to 1 000 ml with water immediately before use. One millilitre of this solution contains 0·01 mg of manganese (as Mn).

A-11.3 Procedure

Accurately weigh about 25 g of the polyphosphoric acid sample and transfer it to a 250 ml beaker and dissolve in about 40ml of water. To this solution, add 10-15 ml of phosphoric acid (85 percent) (*see* **A-11.2.1**) and 0.6 to 0.8 g of potassium periodate. Heat the solution to boiling. Boil for 20 min. Cool the solution to room temperature. Transfer the solution to a 100ml Nessler cylinder. Make up to the mark with distill water and mix well. Prepare a standard by mixing 1 ml of standard manganese solution B (*see* **A-11.2.5**) with 40 ml of water and 10-15 ml of the phosphoric acid (85 percent) (*see* **A-11.2.1**) and 0.6-0.8 g of potassium iodate. Any pink colour produced in the test sample should not be deeper than that of the standard when compared in Nessler cylinders.

A-11.4 Alternative Method

Manganese may alternatively be estimated by instrumental method as **A-18.**

A-12 DETERMINATION OF MOLYBDENUM

A-12.1 Weigh 2.0 g of the material to the nearest 0.01 g in a small glass beaker. Transfer quantitatively to a 500 ml volumetric flask and make up to the mark with distilled water. Mix and use the sample solution for testing of Molybdenum by Atomic Absorption Spectroscopy as described in IS 3025 (Part 2). Multiply the result obtained by 250 to get Molybdenum (as Mo) content in the material.

A-12.2 Alternative Method

Molybdenum may alternatively be estimated by instrumental method as **A-18**.

A-13 DETERMINATION OF MERCURY

A-13.1 Weigh 10 g of the material to the nearest 0.01 g in a small glass beaker. Transfer quantitatively to a 1 000 ml volumetric flask and make up to the mark with distilled water. Mix and use the sample solution for testing of mercury by Atomic Absorption Spectroscopy as described in IS 3025 (Part 48). Multiply the result obtained by 100 to get mercury (as Hg) content in the material.

A-13.2 Alternative Method

Mercury may alternatively be estimated by instrumental method as **A-18.**

A-14 DETERMINATION OF ALUMINIUM

A-14.1 Weigh 0.5 g of the material to the nearest 0.01 g in a small glass beaker. Transfer quantitatively to a 500 ml volumetric flask and make up to the mark with distilled water. Mix and use the sample solution for testing of aluminium by Atomic Absorption Spectroscopy as described in IS 3025 (Part 55). Multiply the result obtained by 1000 to get aluminium (as Al) content in the material.

A-14.2 Alternative Method

Aluminium may alternatively be estimated by instrumental method as **A-18.**

A-15 TEST FOR CALCIUM AND MAGNESIUM

Calcium and magnesium are estimated by the instrumental methods as prescribed in **A-18.**

A-16 DETERMINATION OF SILICA

Sample is used for testing of silica by any of the four methods (Gravimetric method, Molybdosilicate method, Heteropoly blue method, automated method for molybdate reactive silica) given in IS 3025 (Part 35).

A-17 ION CHROMATOGRAPHY FOR CHLORIDES AND SULPHATES

A-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, 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-17.2 Equipments

A-17.2.1 *Anion Guard Column* — A protector of the separator column.

A-17.2.2 *Anion Separator Column* — Suitable for selective separation of ions under analysis.

A-17.2.3 *Anion Suppressor Device* — Anion micromembrane suppressor is used to analyse the data

Detector: Conductivity detector.

A-17.2.4 *Software* — Software suitable for control of various operating parameters, receiving inputs and analysis of all data.

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-17.3 Reagents and Standards

A-17.3.1 Glass or polyethylene sample bottles.

A-17.3.2 Distilled water or deionized water free from the anions of interest.

A-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, 0.2856 g of sodium bicarbonate and 0.3816g of sodium carbonate is dissolved in 2L of water.

Micromembrane suppressor solution: (0.025 N of sulphuric acid)-Dilute 2.8 ml of conc. Sulphuric acid in 4 L of water

A-17.4 Standard Solutions

Chloride:	Dissolve NaCl, 1.6485 g in 1 L of reagent water
Sulphate:	Dissolve 1.81 g of potassium sulphate in 1 L of reagent water

A-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 0.1-1.0 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-17.6 Procedure

Dissolve between 1 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 (0.1-1.0 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 and deviation of retention force shall not exceed ±10 percent of RT of calibration. 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 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-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.

A-18 DETERMINATION OF LEAD, IRON, CALCIUM, MAGNESIUM, MANGANESE, ARSENIC, MOLYBDENUM, ALUMINIUM AND MERCURY BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

A-18.1 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-18.2 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 1 lists the most important spectral interferences at the recommended wavelengths for analysis.

A-18.3 Reagents and Solutions

A-18.3.1. *Nitric Acid (65 Percent) Suprapure*

A-18.3.2 *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 2-5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

A-18.3.3 *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- 18.3.4 *Sample Preparation* — Weigh about 2.5 g polyphosphoric acid sample in a 50 ml volumetric flask and add 1.0 ml Nitric acid and make up the volume with water.

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

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

(*Clause* A-18.2)

Sl No.	Element	Wavelength (nm)	Approx. Achievable Limits		Interfering Elements
			Radial Viewing (µg)	Axial Viewing (µg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Pb	220.353	14	5	Al, Co, Fe, Ti
		283.305	(70)		Cr, Fe
		217.00		(20)	
ii)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-
iii)	Ca	315.887	100	13	Co, Mo
		317.933	26	4	Fe, V
		393.366	0.4	25	V, Zr
		422.673	-	-	V, Mo, Zr
iv)	Mg	279.078	33	19	Fe
		279.553	1	7	Fe
		285.213	4	14	Cr
v)	Mn	257.610	1	0.4	Cr, Fe, Mo, W
		293.305	(20)	8	Al, Cr, Fe, Ti
vi)	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
vii)	Mo	202.031	(30)	(2)	Al, Fe, Ni
		204.597	(50)	(6)	Co, Cr
		281.616			
viii)	Al	167.079	1	2	Fe, Pb
		308.215	100	17	Fe, Mn, OH, V
		396.152	10	6	Cu, Fe, Mo, Zr
ix)	Hg	253.652	2		Cr
		194.164	2		Sn
		184.887	4		Sn

A-18.3.5 *Reagent Blank Solution* — Place 50 ml of nitric acid and 1 000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluorethylene (PTFE) containers should be used. Prior to analysis, make sure that the acid matrix and concentration of the reagent blank solution is the same as in the standard and sample solutions.

A-18.4 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-18.5 Procedure

A-18.5.1 *Calibration*

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**18.3.5**). 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-18.5.2 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, magnesium, manganese, arsenic, molybdenum, aluminium and mercury) 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-18.6 Calculation

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

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

ANNEX B

(*Clause 6*)

SAMPLING OF POLYPHOSPHORIC ACID

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that an ullage of about 5 percent is left after pouring in the sample.

B-1.7 Each sample container shall be sealed air tight after filling, and marked with full details of sampling and the date of sampling.

B-2 SCALE OF SAMPLING

B-2.l Lot

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

B-2.2 Samples shall be tested from each lot separately for judging the conformity of the material to the requirements or the specification. For this purpose 5 containers shall be selected at random from each lot.

NOTE — If the number of containers in the lot is five or less, the number of containers to be selected and the criteria for conformity of the lot to the specification shall be as agreed to between the purchaser and the supplier.

B-2.3 The containers shall be selected at random and to ensure randomness of selection, the following procedure is recommended for use:

Starting from any container in the lot, count them 1,2,……up to *r* and so on, where *r* is the integral part of *N*/5 (*N* being the number of containers in the lot). Every r^{th} container thus counted shall be withdrawn to constitute a sample till the required number of 5 containers is obtained.

B-3 PREPARATION OF TEST SAMPLES

B-3.1 Sampling Tube

The sampling tube shall be made of glass and shall be 20 to 40 mm in diameter and 350 to 750 mm in length. The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For drawing sample, the apparatus is first closed at the top with the thumb or a stopper and lowered till a desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

B-3.1.1 For small containers, the size of the sampling tube may be altered suitably.

B-3.2 From each of the containers selected according to **B-2.3** a small representative portion of the material, about 200 ml, shall be taken out with the help of the sampling tube after thoroughly stirring the acid with the help of a glass rod.

B-3.3 Out of these portions, a small but equal quantity of the material shall be taken out and thoroughly mixed to form a composite sample not less than 600 ml. The composite sample shall be divided into 3 equal parts, one for the purchaser, one for the supplier and the third to be used as a referee sample.

B-3.4 The remaining portion of the material from each container shall be divided into 3 equal parts, each forming an individual sample. One set of individual samples representing the 5 containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.5 All the individual and composite samples shall be transferred to separate bottles. These bottles shall be sealed and labelled with full identification particulars.

B-3.6 The referee samples consisting of composite sample and a set of 5 individual samples shall bear the seals of the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier to be used in the case of a dispute between the two.

B-4 NUMBER OF TESTS

B-4.1 Tests for the determination of polyphosphoric acid shall be performed on each of the 5 individual samples.

B-4.2 Tests for the determination of all other characteristics listed in Table 1 shall be performed on the composite sample only.

B-5 CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples

From the 5 test results for polyphosphoric acid, the mean (x) and range (R) of test results shall be computed (range being defined as the difference between the maximum and minimum values of test results).

B-5.1.1 The lot shall be declared as conforming to the requirements of polyphosphoric acid if the value of the expression (x — 0.6 R) as calculated from the relevant test results is greater than or equal to the minimum value specified in col 3, 4 and 5 of Table 1(depending on the grade of acid under consideration).

B-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 in Table 1.

ANNEX C

(*Foreword*)

EXPERTS WHO MADE SIGNIFICANT CONTRIBUTION TO THE DEVELOPMENT OF THIS STANDARD

COMMITTEE COMPOSITION

Inorganic Chemicals Sectional Committee, CHD 01

Organization	*Representative(s)*
Central Salt and Marine Chemicals Research Institute, Bhavnagar	Dr Kannan Srinivasan (***Chairman)***
Aditya Birla Chemical (I) Ltd, New Delhi	Shri Alok Singh
Alkali Mfrs Association of India, Delhi	Shri K. Srinivasan Shri Subhash Tandon (*Alternate*)
Bhabha Atomic Research Centre, Mumbai	Dr A. V. R. Reddy Dr S. N. Achary (*Alternate*)
Central Drugs Standard Control Organization	Dr Raman Mohan Singh
DGQA, New Delhi	Dr Gurbachan Singh Shri B. S. Tomar (*Alternate*)
Geological Survey of India, Kolkata	Dr D. K. Das Dr Subhas Chandra (*Alternate*)
Grasim Industries Ltd, Nagda	Shri R. S. Baghel Shri Pankaj Gupta (*Alternate*)
Gujarat Alkalies and Chemicals Ltd, Vadodara	Dr Sunil Sinha
Hindustan Lever Ltd, Mumbai	Vrinda Rajwade Shrimati Poornakala (*Alternate* I) Satyamoorthy (*Alternate* II)
In personal capacity	Dr A. N. Bhat
In personal capacity	Dr T. S. Kathpal
Industrial Carbon Pvt.Ltd, Ankleshwar	Shri Rohit Kumar Madhavji Shri Satyan Rohit Kumar (*Alternate*)
Indian Institute of Chemical Technology, Hyderabad	Dr Praveen R. Likhar Dr Rajender Reddy (*Alternate*)
Ministry of Defence (DGQA), Kanpur	Shri R. N. Aparajit
Ministry of Chemicals & Fertilizers	Dr Rohit Misra
National Chemical Laboratory, Pune	Dr Darbha Srinivas Dr Paresh Dhepe (*Alternate*)
National Metallurgical Laboratory, Jamshedpur	Dr Trilochan Mishra Shri Devbrata Mishra (*Alternate*)
National Mineral Development Corporation Ltd, Hyderabad	Shri Rajan Kumar Dr Prashant Sharma (*Alternate*)
National Physical Laboratory, New Delhi	Dr Nahar Singh Dr S. P. Singh (*Alternate*)
National Test House (NR)	Dr Y. C. Nijhawan Dr (Ms) A. Biswas (*Alternate*)
Nirma Chemicals, Ahmedabad	Shri R. A. Joshi, Dr K. C. Pathak (*Alternate*)

Organization	*Representative(s)*
Office of the Development Commissioner (MSME), New Delhi	Shrimati Shimla Meena Shri Santosh Kumar (*Alternate*)
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Headquarters:

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