

भारतीय मानक मसौदा
ट्राइसोडियम फॉस्फेट – विशिष्टि
) आईएस 573 का पांचवां पुनरीक्षण

Draft Indian Standard
TRISODIUM PHOSPHATE — SPECIFICATION
(Fifth Revision of IS 573)

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ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01

Last date of comments: 17th December 2022

FOREWORD

This Indian Standard (Fifth Revision) 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.

This standard was originally published in 1954 and then subsequently revised in 1964, 1973, 1985 and 1992. In the original standard, three types of trisodium phosphate, namely, anhydrous, monohydrate and dodecahydrate were covered. These were retained in the first revision. However, in the second revision, the monohydrate type of trisodium phosphate was deleted as it was not being manufactured in the country. In the second revision, analytical reagent grade of trisodium phosphate was added and a new requirement of free sodium hydroxide was incorporated. During the third revision, a new requirement of free alkalinity was prescribed for dodecahydrate type of trisodium phosphate, deleting the requirements of carbonates and free sodium hydroxide for this type.

In previous revision, new requirements of insolubles, soluble silica and chloride contents were prescribed for trisodium phosphate, dodecahydrate along with the relevant test methods. Also, the methods for determination of carbonate alkalinity, phosphate, carbonate, free sodium hydroxide and total alkalinity were suitably modified.

In this revision, instrumental test methods for the determination of arsenic, chlorides, lead and sulphates have been added as alternate test methods. Also, Packing and Marking clause has been updated. Further, all the amendment have been incorporated.

When stored under ordinary conditions, the dodecahydrate may lose some water of crystallization. Any loss in water will result in an assay of more than 100 percent trisodium phosphate on the basis of the assay of sodium phosphate dodecahydrate but will not appreciably affect determination of the relative amount of other characteristics.

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 shall be the same as that of the specified value in this standard.

1 SCOPE

1.1 This standard prescribes requirements and methods of sampling and test for trisodium phosphate.

2 REFERENCES

The Indian Standards listed 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 Indicated below:

<i>IS No.</i>	<i>Title</i>
264 : 2005	Nitric acid — Specification (<i>third revision</i>)
265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)
915 : 2012/ ISO 1042	Laboratory glassware - One - mark volumetric flasks (<i>third revision</i>)
1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)
2088 : 1983	Methods for determination of arsenic (<i>second revision</i>)
2263: 1979	Methods of preparation of indicator solutions (<i>first revision</i>)
2316 :1990	Methods of preparation of standard solutions for colorimetric and volumetric analysis (<i>second revision</i>)
3025 (Part 2) : 2019/ ISO 11885	Methods of sampling and test (physical and chemical) for water and wastewater: Part 2 determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP - OES) (<i>first revision</i>)
4905 : 2015/ ISO 24153	Random sampling and randomization procedures (<i>first revision</i>)

3 GRADES

The material shall be of the following two grades:

a) Technical (Tech) — For use in detergent mixtures, softening water, prevention of hard boiler scale, tanning of leather, clarifying sugar, laundering, manufacture of paper, in photographic developers, and in kier boiling; and

b) Analytical (AR) — For use as a reagent in laboratory.

4 TYPES

4.1 The technical grade material shall be of two types, namely:

a) Dodecahydrate, and

b) Anhydrous.

4.2 The analytical grade shall be of dodecahydrate type only.

5 REQUIREMENTS

5.1 Description

The material shall be in the form of white crystalline powder or in the form of easily friable small lumps.

5.2 The material shall comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col **6** of the table.

6 PACKING AND MARKING

6.1 Packing

The material shall be packed as agreed to between the purchaser and the supplier.

6.2 Marking

The packages shall be legibly marked with the following information:

- a) Name, grade and type of the material;
- b) Mass of the material;
- c) Indication of the source of manufacture;
- d) Year of manufacture; and
- e) Lot number in code or otherwise to enable the batch of manufacture to be traced from records.

6.2.1 In case of analytical reagent grade, the analysis of the material in respect of the characteristics laid down in col **5** of Table 1 shall also appear on the label.

6.2.2 The packages may also be marked with the Standard Mark.

7 SAMPLING

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

Table 1 Requirements for Trisodium Phosphate
(Clause 5.2)

Sl. No.	Characteristic	Requirement for Grade			Method of Test (Ref. to Cl No. in Annex A)
		(Tech)		(AR)	
(1)	(2)	Dodecahydrate	Anhydrous	Dodecahydrate	(6)
i)	Matter insoluble in water, percent by mass, <i>Max</i>	0.20	0.50	0.01	A-2
ii)	Phosphates (as P ₂ O ₅), percent by mass, <i>Min</i>	17.50	41.0 ¹⁾	18.3	A-3 or A-4 ²⁾
iii)	Carbonates (as Na ₂ CO ₃), percent by mass, <i>Max</i>	-	2.0	-	A-4
iv)	Carbonate alkalinity (as NaOH), percent by mass, <i>Max</i>	3.00	—	—	A-4
v)	Free sodium hydroxide (as NaOH), percent by mass, <i>Max</i>	—	3.2	2.5	A-4
vi)	Total alkalinity (as NaOH), percent by mass, <i>Max</i>	4.5	—	—	A-4
vii)	Soluble iron compounds (as Fe), percent by mass, <i>Max</i>	0.009	0.02	0.001	A-5
viii)	Nitrogen compounds (as N), percent by mass, <i>Max</i>	-	-	0.001	A-6
ix)	Sulphates (as SO ₄), percent by mass, <i>Max</i>	-	-	0.01	A-7 or A-13
x)	Arsenic (as As ₂ O ₃), percent by mass, <i>Max</i>	-	-	0.000 5	A-8 or A-12
xi)	Heavy metals (as Pb), percent by mass, <i>Max</i>	-	-	0.001	A-9 or A-12
xii)	Soluble silica (as SiO ₂), percent by mass, <i>Max</i>	0.1	-	0.05	A-10
xiii)	Chloride (as Cl), percent by pass, <i>Max</i>	0.1	-	0.05	A-11 or A-13

NOTE — The requirements for soluble silica (as SiO₂) and chloride (as Cl) for trisodium phosphate, technical, dodecahydrate grade shall be special requirements only for the material intended to be used for softening water and for prevention of hard boiler scale.

ANNEX A
(Clause 5.2)
METHODS OF TEST FOR TRISODIUM PHOSPHATE

A-1 QUALITY OF REAGENTS

Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS 1070) shall be employed in tests.

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

A-2 DETERMINATION OF MATTER INSOLUBLE IN WATER**A-2.1 Procedure**

Weigh accurately about 10 to 20 g of the material and dissolve in about 150 ml of water. Filter the insoluble matter, if any, through a tared filter paper or a sintered glass crucible (G. No. 4) or a Gooch crucible. Wash the residue thoroughly with water till it is free from all soluble compounds and dry at 105°C to 110°C. Cool in a desiccator and weigh to constant mass.

A-2.2 Calculation

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

where

M_1 = mass in g of the residue, and

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

A-3 DETERMINATION OF PHOSPHATE CONTENT**A-3.1 Outline of the Method**

This method involves the formation of phosphomolybdic acid in a solution free from ammonium salts, followed by its precipitation as the salt of quinoline. Finally the quinoline phosphomolybdate is titrated with sodium hydroxide.

A-3.2 Reagents**A-3.2.1 Quinoline Hydrochloride Solution**

Add 20 ml of purified quinoline to 500 ml of hot water acidified with 25 ml of concentrated hydrochloric acid (*see* IS 265). Cool and dilute to 1000 ml.

The quinoline used shall be purified and distilled according to the following method:

Dissolve the technical grade quinoline in concentrated hydrochloric acid and add excess zinc chloride solution. This precipitates quinoline as a complex salt $[(C_9H_7N)_2.ZnCl_4]$ and in well-defined crystals.

Separate and wash the crystals with cold dilute hydrochloric acid. Regenerate the pure quinoline by sodium hydroxide solution. Dry and distil to obtain yield pure and distilled quinoline.

A-3.2.2 Citro-Molybdate Reagent

Prepare the citro-molybdate reagent according to the following procedure:

a) Dissolve 150 g of sodium molybdate ($\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$) in 400 ml of water.

b) Dissolve 250 g of citric acid in 250 to 300 ml of water and 280 ml of concentrated hydrochloric acid (*see* IS 265). To this pour with stirring the solution prepared in (a), cool and filter through a filter pad. A slight greenish colour is obtained on mixing, which may deepen when exposed to sunlight. Add in drops a 0.5 percent (*m/v*) solution of potassium bromate to discharge the colour. Store the solution in coloured, air-tight, stoppered glass bottles in the dark.

A-3.2.3 Mixed Indicator Solution

Mix 3 volumes of alcoholic phenolphthalein solution and 1 volume of alcoholic thymol blue solution (*see* Table 3 of IS 2263).

A-3.2.4 Standard Sodium Hydroxide Solutions

Carbonate-free, 0.5 N and 0.1 N (*see* IS 2316).

A-3.2.5 Standard Hydrochloric Acid — 0.5 N and 0.1 N.

A-3.3 Procedure

Weigh accurately about 2 g of the material into a 250 ml measuring flask and add 50 ml of carbon dioxide-free water. Boil the solution gently for 30 min. Cool to room temperature and make up the volume to 250 ml with carbon dioxide-free water. Filter the solution through a filter paper discarding the first 15 ml. Transfer 25 ml of the filtered solution by means of a pipette into a 250 ml beaker. Add 50 ml of citro-molybdate reagent and heat to boiling. Add 5 drops of quinoline hydrochloride solution with constant stirring during the addition. Again heat to boiling and add quinoline hydrochloride solution drop by drop with constant stirring until 2 ml have been added. To the gently boiling solution, add quinoline hydrochloride solution, few millilitres at a time with constant stirring until a total of 60 ml has been added. A coarsely crystalline precipitate is produced. Allow the beaker to stand on a hot-plate for 15 min and then cool to room temperature. Filter through a filter paper on pulp pad employing suction and wash the flask, precipitate and the filter paper with cold water until they are free from acid.

Transfer the filter paper and the precipitate to the original flask and rinse the funnel with water into the flask. If necessary, wipe the funnel with a small piece of damp filter paper to ensure complete removal of the precipitate and place the paper in the flask. Dilute to about 100 ml with water. Stopper the flask and shake it vigorously until the pulp and the precipitate are completely disintegrated. Remove the stopper and wash it with water, returning the washings to the flask. From a burette add 50 ml of 0.5 N standard sodium hydroxide solution, shaking the flask during addition. Shake vigorously until all the precipitate dissolves (*see* Note). Add 1 ml of mixed indicator solution and titrate the excess of sodium hydroxide solution with 0.5 N hydrochloric acid until the colour changes from violet to green-blue and then sharply to yellow.

NOTE — Examine the disintegrated paper pulp carefully for specks of undissolved precipitate which sometimes dissolve in excess of sodium hydroxide but with difficulty.

A-3.2.1 Carry out a blank determination using all reagents, without the sample and using exactly 0.1 N standard sodium hydroxide solution and 0.1 N standard hydrochloric acid instead of 0.5 N acid and 0.5 N alkali.

A-3.3 Calculation

$$\text{Phosphates (as P}_2\text{O}_5\text{), percent by mass} = \frac{0.136}{M} \left[(V_1 - V_2) - \frac{(V_3 - V_4)}{5} \right]$$

where

V_4 = volume in ml of 0.5 N sodium hydroxide solution used with the sample,

V_2 = volume in ml of 0.5 N hydrochloric acid used with the sample,

V_3 = volume in ml of 0.1 N sodium hydroxide used in the blank,

V_4 = volume in ml of 0.1 N hydrochloric acid used in the blank, and

M = mass in g of the material contained in the solution, taken for the precipitation.

A-4 DETERMINATION OF CARBONATE ALKALINITY AND PHOSPHATE, CARBONATE, FREE SODIUM HYDROXIDE AND TOTAL ALKALINITY

A-4.1 Outline of the Method

In an aliquot of the solution of the material, total alkali and phosphates are titrated with a standard acid. The acid phosphates formed are precipitated with silver nitrate and the free acid liberated is titrated with alkali which gives the phosphate content. In another aliquot of the solution, carbonates and phosphates are precipitated with barium chloride and free alkali is determined by titration with standard hydrochloric acid. Carbonate alkalinity is then determined from the data obtained.

A-4.2 Reagents

A-4.2.1 Barium Chloride Solution — about 10 percent.

A-4.2.2 Phenolphthalein Indicator Solution — 0.1 g in 100 ml of 60 percent rectified spirit.

A-4.2.3 Standard Hydrochloric Acid — 0.1 N.

A-4.2.4 Methyl Orange Indicator Solution — 0.01 g in 100 ml of water.

A-4.2.5 Silver Nitrate Solution — about 5 percent.

A-4.2.6 Benzyl Alcohol

A-4.2.7 Methyl Red Indicator Solution — 0.15 g in 500 ml of water.

A-4.2.8 Standard Sodium Hydroxide Solution — 0.1 N.

A-4.3 Procedure

A-4.3.1 Weigh accurately about 5 g of the material in case of dodecahydrate grade and 2.5 g of the material in case of anhydrous grade and dissolve it in water. Filter off insolubles, if any, and make up the filtrate and the washings to 250 ml in a volumetric flask.

A-4.3.2 Transfer 25 ml aliquot of the solution prepared in **A-4.3.1** to a conical flask and add 40 to 50 ml of barium chloride solution so that all the phosphates and the carbonates are precipitated off. Add 0.2 ml of phenolphthalein indicator and titrate with standard hydrochloric acid to colourless end point (V_1).

A-4.3.3 Transfer 25 ml aliquot of the solution prepared in **A-4.3.1** to a 250 ml beaker, add 1 drop of methyl orange indicator and titrate with standard hydrochloric acid to light pink end point (V_2).

A-4.3.4 Add sufficient silver nitrate solution to precipitate all the phosphate (35 to 40 ml are usually sufficient), 2 ml of benzyl alcohol to coagulate all the precipitate and 0.5 ml of methyl red indicator. Titrate the liberated nitric acid with standard sodium hydroxide solution. The end point shall be from red to yellow (V_3).

A-4.3 Calculation

$$\text{Phosphate (as P}_2\text{O}_5\text{), percent by mass} = \frac{V_3 \times 3.55}{M}$$

$$\text{Free sodium hydroxide (as NaOH), percent by mass} = \frac{V_1 \times 4.0}{M}$$

$$\text{Carbonates (as Na}_2\text{CO}_3\text{), percent by mass} = \frac{(V_2 - V_3 - V_1) \times 5.30}{M}$$

$$\text{Total alkalinity (as NaOH), percent by mass} = \frac{(V_2 - V_3) \times 4.0}{M}$$

$$\text{Carbonate alkalinity (as NaOH), percent by mass} = \frac{(V_2 - V_3 - V_1) \times 4.0}{M}$$

where

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

A-5 DETERMINATION OF SOLUBLE IRON COMPOUNDS**A-5.1 Outline of the Method**

Iron is determined colorimetrically by measuring the transmittance of reddish-violet colour produced by ferrous and ferric iron with thioglycollic acid at pH 10.

A-5.2 Apparatus**A-5.2.1 Photoelectric Absorptiometer****A-5.2.2 One-Mark Graduated Flasks** — 100 ml capacity (*see* IS 915).**A-5.3 Reagents**

A-5.3.1 Dilute Hydrochloric Acid — approximately 5 N.

A-5.3.2 Sodium Citrate Solution (1 M) or Ammonium Citrate Solution (2 M)

29.4 g of sodium citrate in 100 ml of water or 43.0 g of ammonium citrate in 100 ml of water.

A-5.3.3 Ammonium Hydroxide — approximately 16 N.

A-5.3.4 Thioglycollate Reagent

Add 20 ml of ammonium hydroxide in 30 ml of water, then add a mixture comprising 10 ml of thioglycollic acid and 40 ml of water.

A-5.3.5 Standard Iron Solution

Dissolve 0.702 g of ferrous ammonium sulphate in 10 ml of dilute sulphuric acid [10 percent (v/v)] and dilute with water to 1000 ml. Take 10 ml of this solution and dilute to 100 ml. One millilitre of this solution contains 0.01 mg of iron (as Fe).

A-5.4 Procedure

Take several aliquots of the standard iron solution in one-mark graduated flasks, add to each of these flasks 4 ml of sodium citrate solution or 2 ml of ammonium citrate solution, 5 ml of thioglycollate reagent and 15 ml of ammonium hydroxide, swirling after each addition. Dilute to the mark and mix well. After 10 min, determine the absorbance against a reagent blank at 535 nm using green filter No. 4 and 4 cm cell (use 1-cm cell if the colour is too strong). Draw a calibration curve by plotting concentration (in µg/ml) against absorbance.

A-5.4.1 Weigh accurately about 1 g of the material and dissolve in 20 ml of water. Add 5 ml of dilute hydrochloric acid, stir slowly with a glass rod, cover with watch glass and boil gently for 15 to 20 min. Cool and transfer to a one-mark graduated flask. Add 4 ml of sodium citrate solution or 2 ml of ammonium citrate solution, 5 ml of thioglycollate reagent and 15 ml of ammonium hydroxide. Dilute to the mark and after 10 min determine the absorbance at 535 nm using green filter No. 4 and 4 cm cell (use 1 cm cell if the colour is too strong). Read the concentrations (in µg/ml) of this solution from the calibration curve and calculate total amount of soluble iron present (as Fe).

A-6 DETERMINATION OF NITROGEN COMPOUNDS

A-6.1 Apparatus

A-6.1.1 Nessler Cylinders — 50 ml capacity.

A-6.2 Reagents

A-6.2.1 Sodium Hydroxide Solution — approximately 10 percent.

A-6.2.2 Devarda's Alloy — consisting of copper 50 parts, aluminium 45 parts and zinc 5 parts.

A-6.2.3 Nessler Solution

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

A-6.2.4 Dilute Hydrochloric Acid — same as in **A-5.3.1**.

A-6.2.5 Standard Ammonium Chloride Solution

Dissolve 0.382 g of ammonium chloride in water and make up to exactly 100 ml. Take 10 ml of the solution and dilute to exactly 1000 ml. One millilitre of this solution contains 0.01 mg of nitrogen (as N).

A-6.3 Procedure

Weigh 1.0 g of the material and dissolve in 50 ml of water. Add 20 ml of sodium hydroxide solution, 0.5 g of powdered Devarda's alloy and allow to stand for 2 h in a distillation flask protected from loss or gain of ammonia. Then slowly distil 30 to 35 ml into 5 ml of water containing 1 drop of dilute hydrochloric acid. Transfer to a Nessler cylinder, add 1 ml of sodium acid.

Transfer to a Nessler cylinder, add 1 ml of sodium hydroxide solution and 2 ml of Nessler solution. Make up the volume to 50 ml mark. Carry out a control test in another Nessler cylinder using for comparison 1 ml of standard ammonium chloride solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture.

A-6.3.1 The limit prescribed in Table 1 shall be considered as not having been exceeded if the intensity of the colour produced in the test with the material is not greater than that produced in the control test.

A-7 DETERMINATION OF SULPHATES

A-7.1 Reagents

A-7.1.1 Dilute Hydrochloric Acid — same as in **A-5.2.1**.

A-7.1.2 Barium Chloride Solution — same as in **A-4.2.1**.

A-7.2 Procedure

Weigh accurately about 10 g of the material and dissolve in 100 ml of water and 2 ml of dilute hydrochloric acid. Filter, if necessary; heat the filtrate to boiling; add slowly, with constant stirring, 5 ml of barium chloride solution; and allow to stand overnight. Filter, wash the precipitate with hot water and ignite at about 800°C. Cool and weigh till constant mass is obtained.

A-7.3 Calculation

Sulphates (as SO₄), percent by mass = $41.15 \frac{M_1}{M_2}$
where

M_1 = mass in g of the precipitate, and

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

A-7.4 Alternative Method

Sulphates may alternatively be determined by instrumental test method as prescribed in **A-13**.

A-8 DETERMINATION OF ARSENIC

A-8.1 Procedure

Dissolve 1.0 g of the material in 10 ml of water. Carry out the test for arsenic as prescribed in IS 2088, using for comparison a stain obtained with 0.005 mg of arsenic trioxide (as As_2O_3).

A-8.1.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the length of the stain as well as the intensity of its colour produced in the test with material are not greater than those produced in the control test.

A-8.2 Alternative Method

Arsenic may alternatively be determined by instrumental test method as prescribed in **A-12**.

A-9 DETERMINATION OF HEAVY METALS

A-9.1 Apparatus

A-9.1.1 *Nessler Cylinders* — 50 ml capacity.

A-9.2 Reagents

A-9.2.1 *Acetic Acid* — approximately 33 percent.

A-9.2.2 *Dilute Ammonium Hydroxide* — approximately 10 percent.

A-9.2.3 *Hydrogen Peroxide* — approximately 6 percent.

A-9.2.4 *Potassium Cyanide Solution*

Dissolve 10 g of potassium cyanide in 90 ml of water, add 2 ml of hydrogen peroxide, allow to stand for 24 h and make up to 100 ml with water.

A-9.2.3 *Concentrated Nitric Acid* — See IS 264.

A-9.2.6 *Standard Lead Solution*

Dissolve 1.60 g of lead nitrate $[\text{Pb}(\text{NO}_3)_2]$ in water and make up the solution to 1000 ml exactly. Pipette out 10 ml of the solution and dilute again in a volumetric flask to 100 ml with water. One millilitre of this solution contains 0.1 mg of lead (as Pb).

A-9.2.7 *Sodium Sulphide Solution* — approximately 10 percent.

A-9.3 Procedure

Weigh 7.0 g of the material into a Nessler cylinder and dissolve in 30 ml of hot water. Add 5 ml of acetic acid. Make it alkaline to litmus by gradual addition of dilute ammonium hydroxide and add 1 ml of potassium cyanide solution. Carry out a control test in another Nessler cylinder using 2.0 g of the material, 0.5 ml of standard lead solution and the same quantities of other reagents. Filter both the solutions, if they are turbid, and if the colours of the solutions differ, equalize by the addition of a few drops of a highly diluted solution of burnt sugar or other non-reactive substance. Dilute both the solutions with water and make up the volume of each to 50 ml. Add 2 drops of sodium sulphide solution, mix thoroughly and compare the colours developed in the two cylinders after 5 min.

A-9.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the colour produced in the test with the material is not deeper than that produced in the control test.

A-9.4 Alternative Method

Lead may alternatively be determined by instrumental test method as prescribed in **A-12**.

A-10 DETERMINATION OF SOLUBLE SILICA

A-10.1 Outline of the Method

This colorimetric method depends on forming Molybdi-silicic acid by reaction of silicic acid with ammonium molybdate in the acidic medium. 1, Amino-2 Naphthol-4 Sulphonic acid is added to reduce the molybdi-silicic compound. This method is designed to determine the soluble silica of low range with high accuracy.

A-10.2 Reagents

A-10.2.1 Reducing Agent (1, Amino-2-Naphthol 4, Sulphonic Acid)

Dissolve 0.5 g of 1, Amino-2-Naphthol, 4, Sulphonic acid and 1 g of sodium sulphite in 50 ml of silica free distilled water. Add this solution to 150 ml of another solution containing 30 g of sodium hydrogen sulphite (NaHSO_3) and make up to 200 ml.

NOTE — This mixture has to be stored in dark coloured polythene book. It is advisable to prepare fresh solution once in two weeks or whenever the stock solution turns into dark colour.

A-10.2.2 Hydrochloric Acid — 1:1 (v/v).

Dilute 1 volume of concentrated hydrochloric acid (AR sp. gr. 1.19) with one volume of distilled water.

A-10.2.3 Ammonium Molybdate Solution — 10 percent (m/v).

Dissolve 10 g of ammonium molybdate tetrahydrate in 100 ml of distilled water. Filter this each day before use.

A-10.2.4 Oxalic Acid — 10 percent (m/v).

Dissolve 10 g of oxalic acid dihydrate ($\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) (AR) in 100 ml of distilled water.

A-10.2.5 *Standard Silica Solution*

Fuse 1 g of high purity quartz with 4 g of sodium carbonate (AR Grade) in a platinum crucible at 1000°C. Dissolve the residue in silica free water and dilute to 1000 ml in a volumetric flask. One ml of this solution is equivalent to 1 mg of silica (as SiO_2). Store the solution in polyethylene bottles.

A-10.3 Procedure

Dissolve 5 g of the sample in 250 ml of water. Dilute 10 ml of this solution to 250 ml. Pipette 50 ml of the clear sample solution into a polyethylene container. Add 1 ml of 1:1 hydrochloric acid solution. Add 4 ml of ammonium molybdate solution in quick succession, mix them thoroughly and allow it to settle. Wait for 5 min. Add 5.0 ml of oxalic acid solution and mix well. Allow it to stand for about 2 min. Then add 2 ml of reducing agent, stir well and allow it to stand for 5 min. Carry out a blank test using all the reagents except the sample in the same total volume as in the sample solution test.

A-10.3.1 Measure the absorbance of the sample in the spectrophotometer at 815 nm and read the value, directly from the calibration curve, made by taking different aliquots of standard silica solution. Deduct the silica content obtained in the blank determination from that obtained in the sample determination.

A-11 DETERMINATION OF CHLORIDE

A-11.1 Apparatus

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

A-11.2 Reagent

A-11.2.1 *Nitric Acid* — concentrated.

A-11.2.2 *Silver Nitrate* — 10 percent solution in water.

A-11.2.3 *Standard Chloride Solution*

Dissolve 824.1 mg sodium chloride (AR Grade, dried at 140°C) in chloride free water and dilute to 1000 ml. One millilitre of this solution contains 500 mg of chloride as Cl.

A-11.3 Procedure

Dissolve 1 g of the sample in 20 ml of water and add 3 ml of concentrated nitric acid and 1 ml of 10 percent silver nitrate solution. Dilute to 50 ml in a Nessler cylinder. The turbidity produced shall not be greater than that produced by 2 ml of standard chloride solution for technical grade and 1 ml of AR Grade of material diluted to the same volume with all the reagents.

A-11.4 Alternative Method

Chlorides may alternatively be determined by instrumental test method as prescribed in **A-13**.

A-12 DETERMINATION OF ARSENIC AND LEAD BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD**A-12.1 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-12.2 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.

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

Sl No.	Element	Wavelength (nm)	Approximately 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 Cr, Fe
		283.305	(70)		
		217.00		(20)	
ii)	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

A-12.3 Reagents and Solutions**A-12.3.1 Nitric acid (65 percent) Suprapure****A-12.3.2 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 1000 µg/ml of arsenic and lead in 2-5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

A-12.3.3 *Standard solution*

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

A-12.3.4 *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

A-12.3.5 *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-12.4 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-12.5 Procedure**A-12.5.1** *Calibration*

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**A-12.5.2**). 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-12.5.2 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 arsenic and lead 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-12.6 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).

A-13 ION CHROMATOGRAPHY FOR CHLORIDES AND SULPHATES

A-13.1 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 *pH* of the column. Similarly, in anion exchange chromatography, the stationary phase is cationic in nature. These cations will then separate the anions.

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

A-13.2 Equipment

A-13.2.1 *Anion guard column* — a protector of the separator column.

A-13.2.2 *Anion Separator column* — suitable for selective separation of ions under analysis.

A-13.2.3 *Anion Suppressor device* — Anion micromembrane suppressor is used to analyse the data
Detector: Conductivity detector.

A-13.2.4 *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 1000 µl be used to determine ionic concentration as per instrument manual and practice.

A-13.3 Reagents and Standards

A-13.3.1 *Glass or polyethylene sample bottles.*

A-13.3.2 *Distilled water or deionized water free from the anions of interest.*

A-13.3.3 *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 0.3816 g of sodium carbonate is dissolved in 2 l of water.

A-13.3.4 *Micromembrane suppressor solution* (0.025 N of sulphuric acid)

Dilute 2.8 ml of concentrated Sulphuric acid in 4 l of water

A-13.4 Standard solutions

A-13.4.1 *Chloride* — Dissolve NaCl, 1.6485 g in 1 l of reagent water

A-13.4.2 *Sulphate* — Dissolve 1.81 g of potassium sulphate in 1 l of reagent water

A-13.5 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-13.6 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-13.7 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.

SAMPLING OF TRISODIUM PHOSPHATE

B-1 GENERAL REQUIREMENTS OF SAMPLING

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

B-1.2 Samples shall not be taken at a place exposed to the adverse effects of weather.

B-1.3 The sampling instruments and sample containers shall be clean and dry.

B-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.6 The samples shall be placed in suitable, clean, dry, and air-tight glass or other suitable containers on which the material has no action.

B-1.7 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.8 Each sample container shall be suitably stoppered and sealed air-tight after filling, and marked with full particulars of the material (*see 6.2*), the date of sampling and the lot and batch number.

B-1.9 Samples shall be stored in a cool and dry place.

B-2 SCALE OF SAMPLING

B-2.1 Lot

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

B-2.1.1 Samples shall be tested for each lot for ascertaining the conformity of the material to the requirements of the specification.

B-2.2 The number (n) of containers to be selected at random from the lot shall depend on the size of the lot and shall be in accordance with Table 3.

B-2.3 These containers shall be chosen at random from the lot. For random selection procedures, guidance can be had from IS 4905. In case this standard is not available, the following procedure shall be adopted:

Arrange all the containers in the lot in a systematic manner and starting from any container count them as 1, 2, 3,.... up to r , where r is the integral part of N/n (N and n being the lot size and sample size respectively). Every r^{th} container thus counted shall be drawn to constitute the sample.

Table 3 Number of Containers to be selected from Lots of Different Sizes
(*Clause B-2.2*)

Lot Size <i>N</i> (1)	Sample Size <i>n</i> (2)
Up to 15	3
16 to 40	4
41 to 110	5
111 to 180	6
181 to 300	7
301 to 500	8
501 to 800	9
801 and above	10

B-3 TEST SAMPLES AND REFEREE SAMPLES

B-3.1 From each of the containers selected according to **B-2.3** draw with an appropriate sampling instrument a representative portion of the material, sufficient for carrying out the tests specified under **5**. These shall constitute the individual samples.

B-3.2 From each of the individual samples, a small but equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample.

B-3.3 The material in the individual and composite test samples shall be divided into three equal parts and transfer to separate bottles, sealed and labelled with full identification particulars of the samples (*see B-1.7*). The material in each such bottle shall constitute a test sample. Separate these test samples representing individual and the composite samples into three identical sets of test samples.

B-3.4 Distribution of Test Samples

One set of test samples shall be sent to the purchaser and another to the supplier. The third set of test samples bearing the seals of the purchaser and the supplier shall constitute the referee sample to be used in case of dispute between the purchaser and the supplier, and shall be kept at a place agreed to between the purchaser and the supplier.

B-4 NUMBER OF TESTS

B-4.1 Tests for determination of matter insoluble in water and phosphate shall be carried out on each of the set of test samples representing individual samples.

B-4.2 Tests for determination of the remaining characteristics shall be performed on the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 For individual samples for those characteristics which are tested on individual samples, the mean and range of test results shall be computed as follows:

$$\text{Mean } (X) = \frac{\text{sum of the individual test results}}{\text{number of test results}}$$

Range (*R*) = difference between the maximum and minimum values of test results.

B-5.1.1 For declaring the conformity of the lot to the specified requirement of matter insoluble in water, ($\bar{X} + 0.6 R$) computed from test results shall be less than or equal to the relevant limits prescribed in Table 1.

The conformity criterion for phosphate shall be that $(\bar{X} - 0.4 R)$ computed from test results shall be greater than or equal to the relevant limits prescribed in Table 1.

B-5.2 For Composite Sample

For declaring the conformity of the lot to the requirements of all the characteristics tested on the composite sample (*see B-4.2*), the test results shall comply with the corresponding specified values.