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मानक

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“Step Out From the Old to the New”

IS 573 (1992): Trisodium phosphate [CHD 1: Inorganic Chemicals]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”



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IS 573 : 1992  
(Reaffirmed 2010)

भारतीय मानक  
ट्राइसोडियम फास्फेट — विशिष्टि  
( चौथा पुनरीक्षण )

*Indian Standard*  
**TRISODIUM PHOSPHATE — SPECIFICATION**  
*(Fourth Revision)*

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**BUREAU OF INDIAN STANDARDS**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

June 1992

Price Group 5

**AMENDMENT NO. 1 JULY 1993**  
**TO**  
**IS 573 : 1992 TRISODIUM PHOSPHATE —**  
**SPECIFICATION**  
**( Fourth Revision )**

( Page 2, Table 1 ) — Add the following note at the end of Table 1:

'NOTE — The requirements for soluble silica ( as  $\text{SiO}_2$  ) 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.'

( CHD 003 )

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## FOREWORD

This Indian Standard ( Fourth Revision ) was adopted by the Bureau of Indian Standards, after the draft finalized by the General 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 and 1985. 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 this revision, new requirements of insolubles, soluble silica and chloride contents have been 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 have been suitably modified.

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

# *Indian Standard*

## TRISODIUM PHOSPHATE — SPECIFICATION

### ( *Fourth Revision* )

#### 1 SCOPE

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

#### 2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

| <i>IS No.</i> | <i>Title</i>   |
|---------------|--|
| 264 : 1976    | Nitric acid ( <i>second revision</i> )   |
| 265 : 1987    | Hydrochloric acid ( <i>third revision</i> )  |
| 915 : 1975    | One-mark volumetric flask ( <i>first revision</i> )  |
| 1070 : 1977   | Water for general laboratory use ( <i>second revision</i> )  |
| 2088 : 1983   | Methods for determination of arsenic ( <i>second revision</i> )  |
| 2263 : 1979   | Methods of preparation of indicator solutions for volumetric analysis ( <i>first revision</i> )                  |
| 2316 : 1990   | Methods of preparation of standard solutions for colorimetric and volumetric analysis ( <i>second revision</i> ) |
| 4905 : 1968   | Methods for random sampling  |

#### 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)          |   |
|        |   | Dodecahydrate         | Anhydrous          | Dodecahydrate |   |
| (1)    | (2)   | (3)                   | (4)                | (5)           | (6)                                       |
| i)     | Matter insoluble in water, percent by mass, <i>Max</i>                        | 0.20                  | 0.50               | 0.01          | A-2                                       |
| ii)    | Phosphates (as P <sub>2</sub> O <sub>3</sub> ), percent by mass, <i>Min</i>   | 17.50                 | 41.0 <sup>1)</sup> | 18.3          | A-3 or A-4 <sup>2)</sup>                  |
| iii)   | Carbonates (as Na <sub>2</sub> CO <sub>3</sub> ), 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 <sub>4</sub> ), percent by mass, <i>Max</i>                  | -                     | -                  | 0.01          | A-7                                       |
| X)     | Arsenic (as As <sub>2</sub> O <sub>3</sub> ), percent by mass, <i>Max</i>     | -                     | -                  | 0.000 5       | A-8                                       |
| xi)    | Heavy metals (as Pb), percent by mass, <i>Max</i>                             | -                     | -                  | 0.001         | A-9                                       |
| xii)   | Soluble silica (as SiO <sub>2</sub> ), 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                                      |

<sup>1)</sup>On dry ??? at 400°C.

<sup>2)</sup>The method prescribed at A-4 is recommended for routine analysis, in case of dispute, the method prescribed at A-3 shall be referee method.



## 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: 1977) 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

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

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-3.0 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-3.1 Reagents

###### A-3.1.1 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 : 1987). Cool and dilute to 1 000 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  $[(C_9H_7N)_2 \cdot ZnCl_4]$  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-3.1.2 Citro-Molybdate Reagent

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

- a) Dissolve 150 g of sodium molybdate ( $Na_2MoO_4 \cdot 2H_2O$ ) 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 : 1987). 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-3.1.3 Mixed Indicator Solution

Mix 3 volumes of alcoholic phenolphthalein solution and 1 volume of alcoholic thymol blue solution (*see* Table 3 of IS 2263: 1979).

###### A-3.1.4 Standard Sodium Hydroxide Solutions

Carbonate-free, 0.5 N and 0.1 N (*see* IS 2316: 1968).

###### A-3.1.5 Standard Hydrochloric Acid — 0.5 N and 0.1 N.

##### A-3.2 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 minutes. 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 minutes 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

Phosphates (as  $P_2O_5$ ), 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-4.0 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 is 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-4.1 Reagents

**A-4.1.1 Barium Chloride Solution** — about 10 percent.

**A-4.1.2 Phenolphthalein Indicator Solution** — 0.1 g in 100 ml of 60 percent rectified spirit.

**A-4.1.3 Standard Hydrochloric Acid** — 0.1 N.

**A-4.1.4 Methyl Orange Indicator Solution** — 0.01 g in 100 ml of water.

**A-4.1.5 Silver Nitrate Solution** — about 5 percent.

**A-4.1.6 Benzyl Alcohol**

**A-4.1.7 Methyl Red Indicator Solution** — 0.15 g in 500 ml of water.

**A-4.1.8 Standard Sodium Hydroxide Solution** — 0.1 N.

### A-4.2 Procedure

**A-4.2.1** 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. Filler off insolubles, if any, and make up the filtrate and the washings to 250 ml in a volumetric flask.

**A-4.2.2** Transfer 25 ml aliquot of the solution prepared in **A-4.2.1** 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-4.2.3** Transfer 25 ml aliquot of the solution prepared in **A-4.2.1** 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-4.2.4** 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

Phosphate (as  $P_2O_5$ ), percent by mass

$$= \frac{V_3 \times 3.55}{M}$$

Free sodium hydroxide (as NaOH), percent by mass

$$= \frac{V_1 \times 4.0}{M}$$

Carbonates (as  $Na_2CO_3$ ), percent by mass

$$= \frac{(V_2 - V_3 - V_1) \times 5.30}{M}$$

Total alkalinity (as NaOH), percent by mass

$$= \frac{(V_2 - V_3) \times 4.0}{M}$$

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-5.0 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-5.1 Apparatus

#### A-5.1.1 Photoelectric Absorptiometer

**A-5.1.2 One-Mark Graduated Flasks** — 100-ml capacity (see IS 915 : 1975).

### A-5.2 Reagents

**A-5.2.1 Dilute Hydrochloric Acid** — approximately 5 N.

**A-5.2.2 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-5.2.3 Ammonium Hydroxide** — approximately 16 N.

#### A-5.2.4 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-5.2.5 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 1 000 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-5.3 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 minutes, 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  $\mu\text{g/ml}$ ) against absorbance.

**A-5.3.1** 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 minutes. 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 minutes 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  $\mu\text{g/ml}$ ) 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-5.2.1.

#### 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 1 000 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 hours 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.

## IS 573 : 1992

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-4.1.1**.

### 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  $\text{SO}_4$ ), 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-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 2088:1983, using for comparison a stain obtained with 0.005 mg of arsenic trioxide (as  $\text{As}_2\text{O}_3$ ).

**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-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 hours and make up to 100 ml with water.

**A-9.2.3 Concentrated Nitric Acid** — See IS 264 : 1976.

**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 1 000 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 minutes.

**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-10 DETERMINATION OF SOLUBLE SILICA

### A-10.0 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-10.1 Reagents****A-10.1.1 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 ( $\text{NaHSO}_3$ ) 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-10.1.2 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-10.1.3 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-10.1.4 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-10.1.5 Standard Silica Solution**

Fuse 1 g of high purity quartz with 4 g of sodium carbonate (AR Grade) in a platinum crucible at  $1000^\circ\text{C}$ . Dissolve the residue in silica free water and dilute to 1 000 ml in a volumetric flask. One ml of this solution is equivalent to 1 mg of silica (as  $\text{SiO}_2$ ). Store the solution in polyethylene bottles.

**A-10.2 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 minutes. Add 5.0 ml of oxalic acid solution and mix well. Allow it to stand for about 2 minutes. Then add 2 ml of reducing agent, stir well and allow it to stand for 5 minutes. Carry out a blank test using all the reagents except the sample in the same total volume as in the sample solution test.

**A-10.2.1** 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  $140^\circ\text{C}$ ) in chloride free water and dilute to 1 000 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.

## ANNEX B (Clause 7)

### SAMPLING OF TRISODIUM PHOSPHATE

#### B-1 GENERAL REQUIREMENTS OF SAMPLING

**B-1.0** In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

**B-1.1** Samples shall not be taken at a place exposed to the adverse effects of weather.

**B-1.2** The sampling instruments and sample containers 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 or other suitable containers on which the material has no action.

**B-1.6** The sample containers shall be of such a size that they are almost completely filled by the sample.

**B-1.7** 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-1.8** 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 2.

**B-2.3** These containers shall be chosen at random from the lot. For random selection procedures, guidance can be had from IS 4905:1968. 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 2 Number of Containers to be Selected from Lots of Different Sizes**  
(Clause B-2.2)

| Lot Size      | Sample Size |
|---------------|-------------|
| $N$<br>(1)    | $n$<br>(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 transferred 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:

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

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.

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