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Indian Standard

DISODIUM PHOSPHATE, ANHYDROUS—
SPECIFICATION

(Second Revision)

UDC 661.833 : 455.2

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

December 1993
FOREWORD

This Indian Standard ( Second 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.

Disodium phosphate is also known as exsiccated sodium phosphate, phosphate of soda, sodium phosphate dibasic and disodium hydrogen ortho-phosphate.

Disodium phosphate, anhydrous is used in the manufacture of detergents and boiler compounds. It is also used in soldering and brazing, in ceramics, for fire proofing of wood and textiles, in pharmaceutical industry, and in analytical chemistry as a buffer for colorometric pH determinations.

This standard was originally published in 1954 and subsequently revised in 1969 incorporating pure and exsiccated grades of disodium phosphate. In this revision, exsiccated grade has been replaced by technical grade of the material. In the technical grade the requirements for chloride, sulphate, arsenic and heavy metals have been deleted as these requirements are not critical for this grade of the material.

The committee responsible for 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.
AMENDMENT NO. 1 JANUARY 1998
TO
IS 567:1993 DISODIUM PHOSPHATE,
ANHYDROUS—SPECIFICATION
(Second Revision)
(Page 5, clause A-11.2.5, line 4) — Substitute '1 000 ml' for '100 ml'.

(CHD 3)
Reprography Unit, BIS, New Delhi, India
Indian Standard
DISODIUM PHOSPHATE, ANHYDROUS –
SPECIFICATION
(Second Revision)

1 SCOPE
This standard prescribes requirements and methods of sampling and test for disodium phosphate, anhydrous.

2 REFERENCES
The Indian standards listed below are necessary adjuncts to this standard:

<table>
<thead>
<tr>
<th>IS No.</th>
<th>Title</th>
</tr>
</thead>
<tbody>
<tr>
<td>264 : 1976</td>
<td>Nitric acid (second revision)</td>
</tr>
<tr>
<td>265 : 1993</td>
<td>Hydrochloric acid (fourth revision)</td>
</tr>
<tr>
<td>323 : 1959</td>
<td>Rectified spirit (revised)</td>
</tr>
<tr>
<td>1070 : 1992</td>
<td>Reagent grade water (third revision)</td>
</tr>
<tr>
<td>2088 : 1983</td>
<td>Methods for determination of arsenic (second revision)</td>
</tr>
<tr>
<td>4905 : 1968</td>
<td>Methods for random sampling</td>
</tr>
</tbody>
</table>

3 GRADES
The material shall be of the following three grades:

a) Technical (Tech),
b) Pure, and
c) Analytical Reagent (AR).

4 REQUIREMENTS

4.1 Description
The material shall be in the form of white, hygroscopic powder.

4.2 The material shall also comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in Annex A. References to the relevant clauses of Annex A are given in col 6 of Table 1.

<table>
<thead>
<tr>
<th>SI No.</th>
<th>Characteristic</th>
<th>Requirements for Grade</th>
<th>Method of Test (Ref to Cl No. in Annex A)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1)</td>
<td>(2)</td>
<td>Tech</td>
<td>Pure</td>
</tr>
<tr>
<td>i)</td>
<td>Assay (as Na₂HPO₄), on dry basis, percent by mass, Min</td>
<td>95.0</td>
<td>99.5</td>
</tr>
<tr>
<td>ii)</td>
<td>pH [of 2 percent (m/v) aqueous solution]</td>
<td>9.1 to 9.2</td>
<td>9.1 to 9.2</td>
</tr>
<tr>
<td>iii)</td>
<td>Matter insoluble in water, percent by mass, Max</td>
<td>0.50</td>
<td>0.01</td>
</tr>
<tr>
<td>iv)</td>
<td>Loss on drying, percent by mass, Max</td>
<td>2.0</td>
<td>0.25</td>
</tr>
<tr>
<td>v)</td>
<td>Chlorides (as Cl), percent by mass, Max</td>
<td>—</td>
<td>0.002</td>
</tr>
<tr>
<td>vi)</td>
<td>Total nitrogen (as N), percent by mass, Max</td>
<td>—</td>
<td>0.001</td>
</tr>
<tr>
<td>vii)</td>
<td>Sulphates (as SO₄), percent by mass, Max</td>
<td>—</td>
<td>0.01</td>
</tr>
<tr>
<td>viii)</td>
<td>Arsenic (as As₂O₃), ppm, Max</td>
<td>—</td>
<td>3</td>
</tr>
<tr>
<td>ix)</td>
<td>Heavy metals (as Pb), percent by mass, Max</td>
<td>—</td>
<td>0.001</td>
</tr>
<tr>
<td>x)</td>
<td>Iron (as Fe), percent by mass, Max</td>
<td>0.01</td>
<td>0.001</td>
</tr>
</tbody>
</table>
5 PACKING AND MARKING

5.1 Packing

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

5.2 Marking

The packages shall be legibly and indelibly marked with the following information:

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

5.2.1 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 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.

ANNEX A

(Clause 4.2)

ANALYSIS OF DISODIUM PHOSPHATE, ANHYDROUS

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS 1070:1992) 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 ASSAY

A-2.1 Reagents

A-2.1.1 Standard Hydrochloric Acid — 0.5 N.

A-2.1.2 Bromocresol Green

Dissolve 0.1 g of bromocresol green in 100 ml of rectified spirit (see IS 323:1959).

A-2.1.3 Methyl Red Indicator Solution

Dissolve 0.3 g of water soluble methyl red in water and dilute to 1 000 ml.

A-2.2 Procedure

Weigh accurately about 2 g of the material dried as under A-5 and dissolve in 100 ml of water. Titrate the solution with standard hydrochloric acid using a mixture of equal parts of bromocresol green and methyl red as indicator until the grey colour indicative of pH 4.5 is obtained.

A-2.3 Calculation

\[ \text{Disodium phosphate (Na}_2\text{HPO}_4\text{), percent by mass} = \frac{14\cdot189 VN}{M} \]

where

\[ V = \text{volume in ml of standard hydrochloric acid required for the titration,} \]

\[ N = \text{normality of the standard hydrochloric acid, and} \]

\[ M = \text{mass in g of the material taken for the test.} \]

A-3 DETERMINATION OF pH

A-3.1 Procedure

Dissolve 2.0 g of the material in 100 ml of freshly boiled and cooled water and determine the pH of the solution by means of a suitable pH-meter using glass electrodes.

A-4 DETERMINATION OF MATTER INSOLUBLE IN WATER

A-4.1 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 Qooch crucible. Wash the residue thoroughly with water till it is free from all soluble matter and dry at a temperature of 105 to 110°C. Cool the residue in a desiccator and weigh to constant mass.

A-4.2 Calculation

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

where

\[ M_1 = \text{mass in g of the residue, and} \]

\[ M_2 = \text{mass in g of the material taken for the test.} \]
A-5 DETERMINATION OF LOSS ON DRYING

A-5.1 Procedure
Weigh accurately about 10 g of the material in a tared porcelain basin of 6 to 8 cm diameter and 1 to 3 cm depth. Dry the contents of the basin at 105° ± 2°C and weigh to constant mass.

A-5.2 Calculation
Loss on drying, percent by mass = \(100 \times \frac{M_1 - M_2}{M_1}\)

where

\[ M_1 = \text{mass in g of the material taken for the test, and} \]

\[ M_2 = \text{mass in g of the dried material.} \]

A-6 DETERMINATION OF CHLORIDES

A-6.1 Apparatus
A-6.1.1 Nessler Cylinders — 50-ml capacity.

A-6.2 Reagents
A-6.2.1 Concentrated Nitric Acid — see IS 264 : 1976.

A-6.2.2 Standard Chloride Solution
Dissolve 1.649 g of sodium chloride in water and make up the volume to 1 000 ml. Pipette out 10 ml of the solution, dilute with water and make up the volume to 100 ml. One millilitre of this solution contains 0.1 mg of chloride (as Cl).

A-6.2.3 Silver Nitrate Solution — Approximately 5 percent (m/v).

A-6.3 Procedure
A-6.3.1 Dissolve 10.0 g of the material in 10 ml of water, add 2 ml of concentrated nitric acid and 1 ml of silver nitrate solution in a Nessler cylinder. Make up the solution to 50-ml mark. Carry out a control test in the other Nessler cylinder using 2 ml of standard chloride solution in case of Pure Grade and 0.5 ml in case of AR Grade material and the same quantities of other reagents in the same total volume of the reaction mixture. Stir both the solutions with glass rods and compare the opalescence produced in the two cylinders after 5 minutes.

A-7 DETERMINATION OF TOTAL NITROGEN COMPOUNDS

A-7.1 Apparatus
A-7.1.1 Nessler Cylinders — 50-ml capacity.

A-7.2 Reagents
A-7.2.1 Sodium Hydroxide Solution — Approximately 10 percent (m/v).

A-7.2.2 Devardas Alloy — Consisting of copper 50 parts, aluminium 45 parts and zinc 5 parts.

A-7.2.3 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-7.2.4 Dilute Hydrochloric Acid — 5 N.

A-7.2.5 Standard Ammonium Chloride Solution
Dissolve 0.382 g of ammonium chloride in water and make up to 100 ml. Take 10 ml of the solution and dilute to exactly 1 000 ml. One millilitre of this solution is equivalent to 0.01 mg of nitrogen (as N).

A-7.3 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 Devardas alloy and allow to stand for 2 hours in the distillation flask protected from loss or gain of ammonia. Then, slowly distill 40 to 45 ml into 5 ml of water containing 1 drop of dilute hydrochloric 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-7.3.1 The limits 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-8 DETERMINATION OF SULPHATES

A-8.1 Reagents

A-8.1.1 Dilute Hydrochloric Acid — 5 N.

A-8.1.2 Barium Chloride Solution — 10 percent (m/v).

A-8.2 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 to constant mass.

A-8.3 Calculation

Sulphates (as $SO_4$), percent by mass =

\[
\frac{41 \cdot 15 \times M_1}{M_2}
\]

where

- $M_1$ = mass in g of the precipitate, and
- $M_2$ = mass in g of the material taken for the test.

A-9 DETERMINATION OF ARSENIC

A-9.1 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 0.003 mg of arsenic trioxide (as $As_2O_3$).

A-9.1.1 The limits 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 the material are not greater than those produced in the control test.

A-10 DETERMINATION OF HEAVY METALS (as Pb)

A-10.1 Apparatus

A-10.1.1 Nessler Cylinders — 50-ml capacity.

A-10.2 Reagents

A-10.2.1 Acetic Acid — approximately 33 percent (m/v).

A-10.2.2 Dilute Ammonium Hydroxide — approximately 10 percent (m/m).

A-10.2.3 Hydrogen Peroxide Solution — approximately 6 percent (m/m).

A-10.2.4 Potassium Cyanide Solution

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

A-10.2.5 Concentrated Nitric Acid — See IS 264: 1976.

A-10.2.6 Standard Lead Solution

Dissolve 1.60 g of lead nitrate [Pb (NO$_3$)$_2$] in water and make up the volume to 1 000-ml mark. 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-10.2.7 Sodium Sulphide Solution — approximately 10 percent (m/v).

A-10.3 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 the other Nessler cylinder using 2.0 g of the material, 0.5 ml of standard lead solution in case of Pure Grade and 0.1 ml in case of AR Grade of the material, 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-10.3.1 The limits 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-11 DETERMINATION OF IRON

A-11.0 Outline of the Method

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

A-11.1 Apparatus

A-11.1.1 Photoelectric Absorptiometer

A-11.2 Reagents

A-11.2.1 Dilute Hydrochloric Acid — approximately 5 N.

A-11.2.2 Sodium Citrate Solution — 1 M.

or

Ammonium Citrate Solution — 2 M.

A-11.2.3 Ammonium Hydroxide — approximately 16 N.
A-11.2.4 Thioglycollate Reagent
Add 20 ml of ammonium hydroxide to 30 ml of water, and then add a mixture of 10 ml of thioglycollic acid and 40 ml of water.

A-11.2.5 Standard Iron Solution
Dissolve 0.491 g of ferrous ammonium sulphate \([\text{FeSO}_4\cdot\text{(NH}_4\text{)}_2\cdot\text{SO}_4\cdot6\text{H}_2\text{O}]\) in water, add 10 ml of dilute sulphuric acid (10 percent v/v). Dilute to exactly 100 ml in a volumetric flask. One millimetre of this solution contains 0.1 mg of iron (as Fe\(_2\)O\(_3\)).

A-11.3 Procedure
Weigh accurately 1 g of the material into a 150-ml beaker. Add 30 ml of water and 5 ml of dilute hydrochloric acid. Introduce a glass rod, cover with a watch glass and boil gently for 15 to 20 minutes. Cool and transfer to a 100-ml volumetric flask and dilute to about 70 ml. Place 5 ml of dilute hydrochloric acid and 65 ml of water in a second flask as blank.

A-11.3.1 To each 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, swirling after each addition. Dilute to the mark and mix well.

A-11.3.2 Balance the absorptiometer at blank using green filter No. 4 and 4-cm cells. (Use 1-cm if the colour is too strong). Find the reading with the sample solution. Read off milligram iron from the standard calibration graph (see A-11.3.3).

A-11.3.3 Standard Calibration Graph
Pipette out known volumes of standard iron solution into each of 100-ml volumetric flasks containing 5 ml of dilute hydrochloric acid. Proceed as in A-11.3.1. Suitable aliquots are 0, 1.0, 1.5, 2.0, 2.5 and 3.0 ml. Take the readings. Plot graph with milligram iron as ordinates and absorptiometer readings as abscissae. Draw a line through the points and extend through the origin. Label the graph with particulars of filters and cells used.

A-11.4 Calculation
Iron (as Fe\(_2\)O\(_3\)), percent by mass = 

where 

\[ M_1 = \text{mass in mg of iron (Fe}_2\text{O}_3\) read off from the graph, and} \]

\[ M = \text{mass in g of the material taken for the test.} \]

ANNEX B
(Clause 6.1)

SAMPLING OF DISODIUM PHOSPHATE, ANHYDROUS

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 in an exposed place.

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

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 package selected for the sampling shall be mixed as thoroughly as possible by suitable means.

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

B-1.6 Each sample container shall be suitably stoppered and scaled air-tight after filling and marked with full particulars of the material (see 5.2) and the date of sampling.

B-1.7 Samples shall be stored in a cool and dry place.

B-2 SCALE OF SAMPLING

B-2.1 Lot
All the packages in a single consignment of disodium phosphate, anhydrous 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 groups of packages in each batch shall constitute separate lots.

B-2.2 For ascertaining the conformity of the material in a lot to the requirements of this specification, samples shall be tested for each lot separately. The number of packages to be selected at random from lots of different sizes shall be as given in Table 2.
Table 2 Scale of Sampling

<table>
<thead>
<tr>
<th>Lot Size</th>
<th>Sample Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>( N )</td>
<td>( n )</td>
</tr>
<tr>
<td>(1)</td>
<td>(2)</td>
</tr>
<tr>
<td>3 to 25</td>
<td>3</td>
</tr>
<tr>
<td>26 to 100</td>
<td>4</td>
</tr>
<tr>
<td>101 to 300</td>
<td>5</td>
</tr>
<tr>
<td>301 to 500</td>
<td>6</td>
</tr>
<tr>
<td>501 to ( \leq 1000 )</td>
<td>7</td>
</tr>
<tr>
<td>( &gt; 1000 )</td>
<td>8</td>
</tr>
</tbody>
</table>

B-2.3 In order to ensure randomness of selection, use shall be made of random number table, but if such a table is not available, the following procedure may be adopted:

Startling from any package, count them as 1, 2, 3,.............. up to \( r \) and so on where \( r \) is the integral part of \( N/n \) (see B-2.2). Every \( r \)th package thus counted shall be drawn to constitute the sample.

B-3 INDIVIDUAL SAMPLES AND COMPOSITE SAMPLES

B-3.1 From each of the packages selected according to B-2.3, a representative portion of the material shall be drawn with the help of appropriate sampling instrument. The amount so withdrawn from each package shall be sufficient for carrying out all the tests specified under B-4, and 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 Each of the individual samples and the composite sample shall be transferred to separate bottles and labelled with full identification particulars.

B-4 NUMBER OF TESTS

B-4.1 Tests for determination of disodium phosphate content and the loss on drying shall be carried out on each of the individual samples.

B-4.2 Tests for the 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 the range of test results shall be computed as follows:

\[
\text{Mean} \ (X) = \frac{\text{sum of individual test results}}{\text{number of test results}}, \quad \text{and} \\
\text{Range} \ (R) = \text{difference between the maximum and the minimum values of test results.}
\]

B-5.1.1 For declaring the conformity of the lot, \( X + 0.6 R \) shall be less than or equal to the maximum specified requirements, and \( X - 0.6 R \) shall be greater than or equal to the minimum specified requirements.

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 the test results shall satisfy the corresponding specified requirements.
ANNEX C

( Foreword )

COMMITTEE COMPOSITION

Composition of General Inorganic Chemicals Sectional Committee, CHD 003

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Ministry of Defence ( DGQA )
Ministry of Defence ( R & D )
Travancore Chemicals & Mfg Co Ltd, Mettur Dam

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Shriram Institute for Industrial Research, Delhi
Union Carbide of India Ltd, Bombay

Geological Survey of India, Calcutta

Director General, BIS ( Ex-officio Member )

Member-Secretary
DR R. K. JHA
Deputy Director ( Chem ), BIS

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Composition of Alums and Phosphates Subcommittee, CHD 03 : 01

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C. D. Thakkar and Co, Calcutta
National Thermal Power Corporation, New Delhi
The Dharamsi Morarji Chemical Co Ltd, Ambernath
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