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IS 10508 (2007): Phosphoric Acid, Food Grade [FAD 8: Food Additives]
Indian Standard

PHOSPHORIC ACID, FOOD GRADE — SPECIFICATION
(First Revision)

ICS 67.220.20
FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

With the increased production of processed foods, manufacturers have started adding a large number of substances, generally in small quantities, to improve the appearance, flavour, texture or storage properties of the processed foods. As impurities in these substances have been found to be harmful, it is necessary to have a strict quality control of these food additives. Phosphoric acid is used as an acidulant and is permitted under the Prevention of Food Adulteration Rules, 1955. This standard would help in checking purity which requires to be checked at the stage of manufacture, for it is extremely difficult (and in many cases impossible) to detect the impurity once these substances are added to the processed foods.

This standard was first published in 1983 based on the then existing JECFA ‘Specification and food chemical codex’ of USA. This standard is being revised taking into consideration the latest publication for phosphoric acid issued by JECFA. In this revision, the limit for nitrates, volatile acids, chlorides and sulphates have been included to align with the international requirements and methods of test also been revised and updated.

Due consideration has also been given to the Prevention of Food Adulteration Rules, 1955 and Standard of Weights & Measures (Packaged Commodities) Rules, 1977. However, this standard is subject to restrictions imposed under these, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2: 1960 ‘Rules for rounding off numerical values (revised)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.
Indian Standard
PHOSPHORIC ACID, FOOD GRADE — SPECIFICATION
(First Revision)

1 SCOPE
This standard prescribes the requirements and methods of sampling and test for phosphoric acid, food grade.

2 REFERENCES
The following standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to 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:

<table>
<thead>
<tr>
<th>IS No.</th>
<th>Title</th>
</tr>
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<tbody>
<tr>
<td>1070:1992</td>
<td>Reagent grade water (third revision)</td>
</tr>
<tr>
<td>1699:1995</td>
<td>Methods of sampling and test for food colours (second revision)</td>
</tr>
<tr>
<td>2491:1998</td>
<td>Food hygiene — General principles — Code of practice (second revision)</td>
</tr>
</tbody>
</table>

3 REQUIREMENTS

3.1 Description
Phosphoric acid shall be a clear, colourless, odourless viscous liquid. It shall be miscible in water and ethanol.

Phosphoric acid is strongly acidic, even in high dilution.

3.2 Test for Phosphate
Neutralize a few millilitres of phosphoric acid and add dilute nitric acid. Then add an equal volume of ammonium molybdate solution and warm. A bright canary-yellow precipitate is obtained which is soluble in dilute ammonia.

3.3 The material shall also conform to the requirements given in Table 1.

3.4 The material shall be processed, packed, stored and distributed under hygienic conditions in licenced premises (see IS 2491).

4 PACKING, STORAGE AND MARKING

4.1 Packing
The material shall be securely packed in well-filled containers with minimum access to air. The containers shall be such as to preclude contamination of the contents with metals or other impurities.

4.2 Storage
The material shall be stored in a cool and dry place so as to avoid excessive exposure to heat.

Table 1 Requirements for Phosphoric Acid
(Clause 3.3)

<table>
<thead>
<tr>
<th>Sl No.</th>
<th>Characteristic</th>
<th>Requirement</th>
<th>Method of Test, Ref to</th>
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<tr>
<td></td>
<td>(1)</td>
<td>(2)</td>
<td>(3)</td>
</tr>
<tr>
<td>1)</td>
<td>Purity as ( H_3PO_4 ), percent by mass, ( Min )</td>
<td>85</td>
<td>A</td>
</tr>
<tr>
<td>2)</td>
<td>Nitrates, ( mg/kg ), ( Max )</td>
<td>5</td>
<td>B</td>
</tr>
<tr>
<td>3)</td>
<td>Volatile acids, ( mg/kg ), ( Max )</td>
<td>10</td>
<td>C</td>
</tr>
<tr>
<td>4)</td>
<td>Chlorides, ( mg/kg ), ( Max )</td>
<td>200</td>
<td>D</td>
</tr>
<tr>
<td>5)</td>
<td>Sulphates percent by mass, ( Max )</td>
<td>0.15</td>
<td>E</td>
</tr>
<tr>
<td>6)</td>
<td>Fluoride, ( mg/kg ), ( Max )</td>
<td>10</td>
<td>F</td>
</tr>
<tr>
<td>7)</td>
<td>Arsenic (as As), ( mg/kg ), ( Max )</td>
<td>2</td>
<td>–</td>
</tr>
<tr>
<td>8)</td>
<td>Lead (as Pb), ( mg/kg ), ( Max )</td>
<td>5</td>
<td>–</td>
</tr>
<tr>
<td>9)</td>
<td>Heavy metals, ( mg/kg ), ( Max )</td>
<td>10</td>
<td>–</td>
</tr>
</tbody>
</table>
4.3 Marking

Each container shall be legibly and indelibly marked with the following information:

- a) Name of the material including the words 'Food Grade';
- b) Name and address of the manufacturer;
- c) Date of manufacture;
- d) Batch or Code number;
- e) Net content when packed;
- f) Phosphoric acid content;
- g) Instruction for storage;
- h) Best before date (Month and Year to be given by the manufacturer); and

4.3.1 BIS Certification Marking

The material may also be marked with the Standard Mark.

4.3.1.1 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

Representative samples of the material shall be drawn according to the method prescribed in 4 of IS 1699.

6 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 experimental results.

ANNEX A

[Table 1, SL No. (i)]

DETERMINATION OF PURITY

A-1 REAGENTS

A-1.1 Thymolphthalein Solution — Dissolve 0.100 g of thymolphthalein in 100 ml of ethanol and filter, if necessary.

A-1.2 Sodium Hydroxide — 0.1 N.

A-2 PROCEDURE

Weigh 1.000 g of phosphoric acid into a glass-stoppered flask, dilute with about 100 ml of water, add 0.5 ml of thymolphthalein solution. Titrate with 1 N sodium hydroxide. Each millilitre of 1 N sodium hydroxide is equivalent to 0.049 g of H₃PO₄.

ANNEX B

[Table 1, SL No. (ii)]

DETERMINATION OF NITRATE

B-1 PROCEDURE

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

ANNEX C

[Table 1, SL No. (iii)]

DETERMINATION OF VOLATILE ACIDS

C-1 PROCEDURE

Dilute 60.05 g of the sample with 75 ml of freshly boiled and cooled water in a distilling flask with a spray tap, and distill 50 ml. To the distillate add phenolphthalein and titrate with 0.1 N sodium hydroxide. Not more than 0.1 ml of 0.1 N sodium hydroxide shall be required for neutralization.
ANNEX D
[Table 1, Sl No. (iv)]
TEST FOR CHLORIDES

D-1 PROCEDURE
Place 1.78 g of the sample in a Nessler tube, dissolve it in about 30 ml of water, and neutralize with dilute nitric acid TS, if the solution is alkaline. Add 6 ml of dilute nitric acid TS and dilute to 50 ml with water. If the use of a sample solution is prescribed, transfer the sample solution into a Nessler tube and dilute to 50 ml with water. Transfer 1 ml of 0.01 N hydrochloric acid into another Nessler tube to serve as the standard, add 6 ml of dilute nitric acid TS, and dilute to 50 ml with water.

If the solution containing the sample is not clear, filter both solutions under the same conditions. Add 1 ml of silver nitrate TS to each solution, mix thoroughly, and allow to stand for 5 min protected from direct sunlight. Compare the turbidity of the two solutions by observing the Nessler tubes from the sides and the tops against a black background. The turbidity of the sample solution shall not exceed that of the standard.

ANNEX E
[Table 1, Sl No. (v)]
TEST FOR SULPHATES

E-1 PROCEDURE
Place 1.25 g of the sample in a Nessler tube, dissolve it in about 30 ml of water, and neutralize with dilute hydrochloric acid TS, if the solution is alkaline. Add 1 ml of dilute hydrochloric acid TS and dilute to 50 ml with water. If the use of a sample solution is prescribed, transfer the sample solution into a Nessler tube and dilute to 50 ml with water. Transfer the prescribed volume of 0.01 N sulphuric acid into another Nessler tube to serve as the standard, add 1 ml of dilute hydrochloric acid TS, and dilute to 50 ml with water.

If the solution containing the sample is not clear, filter both solutions under the same conditions. Add 2 ml of barium chloride TS to each solution, mix thoroughly, and allow to stand for 10 min. Compare the turbidity of the two solutions by observing the Nessler tubes from the sides and the tops against a black background. The turbidity of the sample does not exceed that of the standard.

ANNEX F
[Table 1, Sl No. (vi)]
DETERMINATION OF FLUORIDE

F-1 APPARATUS
Assembly as shown in Fig. 1.

FIG. 1 APPARATUS FOR LIMIT TEST FOR FLUORIDE

F-2 REAGENTS
F-2.1 Phenolphthalein Solution — Dissolve 0.2 g of phenolphthalein in 60 ml of 90 percent ethanol and sufficient water to make 100 ml.

F-2.2 Sodium Hydroxide — 1N.

F-2.3 Sulphuric Acid — Concentrated.

F-2.4 Sodium Fluoride Solution — Containing 50 µg of fluoride.

F-2.5 Hydrochloric Acid — 4 N.

F-2.6 Zirconium Alizarin Solution
Dissolve 0.80 g of zirconium nitrate in water, add a few drops of 4N nitric acid and make up to 100 ml with water. Dissolve 0.10 g of alizarin sulphonate monohydrate in
20 ml of water, make up to 100 ml with ethanol. Mix 1 ml of first solution with 1 ml of second solution and add 18 ml of water. The solution should be clear and the dilution should be freshly prepared.

F-3 PROCEDURE

F-3.1 Place about 500 ml of water in flask A (see Fig. 1), make it alkaline to phenolphthalein solution with 1N sodium hydroxide and heat the water to boiling. Leave open the screw clamp at C.

F-3.2 Determine the appropriate weight of the sample (W, in gram) by the formula W = 50/L, in which L is the fluoride limit, in mg/kg. Accurately weigh the calculated amount of the sample, place it in flask B, and add 10 ml of water to the flask. Add 17 ml of sulphuric acid slowly down the sides of the flask so that it forms a layer under the water. Connect flask B to the apparatus. Place the tip of the condenser into a flask containing 5 ml of water. Mix the contents of flask B, heat to 150°C and slowly shut the screw clamp at C. Regulate the temperature of the solution in the flask B to 150-153°C during the distillation. Continue until 70 ml of distillate have been collected.

F-3.3 Place the distillate in a 100 ml Nessler tube. Place 80 ml of sodium fluoride solution, containing 50 µg of fluorine, in a second Nessler tube. To each tube add 8.5 ml of 4N hydrochloric acid and 2.0 ml of zirconium-alizarin solution and make up to 100 ml with water. Let the tubes stand for 15 min.

F-3.4 The colour of the test solution containing the sample shall not be darker than that of the standard solution.
Bureau of Indian Standards

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Amendments Issued Since Publication

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