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भारतीय मानक मसौदा

डाइकैल्शियम फास्फेट, खाद्य ग्रेड - विशिष्टि

(*आइ एस* 9970 *का पहला पुनरीक्षण*)

Draft Indian Standard

DICALCIUM PHOSPHATE, FOOD GRADE — SPECIFICATION

(*First Revision of IS 9970*)

ICS No. 67.220.20

Food Additives Committee, FAD 08 **Last Date of Comments** : 10 May 2024

FOREWORD

(*Formal clauses would be added later*)

Dicalcium phosphate is used as dough conditioner for manufacture of certain foods and as nutrient supplement. It is permitted under the *Food Safety and Standards (Food Products Standards and Food Additives) Regulation,* 2011.

Chemical Characteristics - Its synonyms are calcium hydrogen phosphate, dibasic calcium phosphate. Chemical names are secondary calcium phosphate, calcium hydrogen orthophosphate, calcium hydrogen phosphate.

Chemical formulae are:

Anhydrous - $CaHPO_4$

Dihydrate – $CaHPO_4.2H_2O$

Molecular masses are:

Anhydrous - 136.06

Dihydrate - 172.09

This standard was first published in 1981. In the preparation of this standard considerable assistance were derived from:

a) Specification for identity and purity of some food additives, FAO Nutrition Meeting Report Series NO. 55 B, FAO/ WHO, Rome, 1976; and
b) Food Chemical Codex 1966. Ed. 1, National Academy of Sciences and National Research Council, Washington DC.

In this revision the following major changes have been made:

a) The requirement for heavy metals has been removed as the limit of lead (contaminant in food colours) is already covered through the standard.
b) The marking requirements have been updated.
c) One amendment issued to the previous version of the standard has been incorporated.

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

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for dicalcium phosphate, food grade.

2 TYPES

The product shall be of following two types:

a) Anhydrous
b) Dihydrate

3 REFERENCES

The following Indian Standards 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:

IS No.	*Title*
IS 1070 : 2023	Reagent grade water − Specification (*fourth revision*)
IS 1699 : 1995	Methods of sampling and test for food colours (*second revision*)

4 REQUIREMENTS

4.1 Description - It shall be white crystals or granules, granular powder or powder.

4.2 Identification

4.2.1 *Positive Test for Calcium* - Dissolve about 0.1 g of the sample by warming with a mixture of 5 ml of dilute hydrochloric acid and 5 ml of water. Add 3.5 ml of ammonia solution dropwise with shaking and then add 5 ml of ammonium oxalate solution. A white precipitate shall form.

4.2.2 *Positive Test for Phosphate* - To 10 ml of warm solution (1 in 100) of the sample containing a slight excess of nitric acid, add 10 ml of ammonium molybdate solution. A yellow precipitate shall form.

4.2.3 *Positive Test for Orthophosphate* - Wet the sample with silver nitrate solution. A yellow colour shall be produced.

4.2.4 *Solubility* - The material shall be sparingly soluble in water and insoluble in ethanol.

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

5 PACKING AND STORAGE

5.1 Packing

The material shall be filled in containers with as little air space as possible. The containers shall be such as to preclude air contamination of the contents with metals or other impurities.

5.2 Storage

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

Table 1 Requirements for Dicalcium Phosphate, Food Grade

(*Clause* 4.3)

Sl. No.	Characteristic	Requirements	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Purity (as $CaHPO_4$) after drying at 200°C for 3 h, percent by mass	98.0 – 102.0	Annex A (A-1)
ii)	Loss on drying, percent by mass, after drying at 200°C for 3 hours, a) Anhydrous, *Max* b) Dihydrate	 2.0 18-22	IS 1699
iii)	Fluoride (as F), mg/kg, *Max*	50.0	Annex A (A-2)
iv)	Lead (as Pb), mg/kg, *Max*	4.0	IS 1699
v)	Arsenic (as As), mg/kg, *Max*	3.0	IS 1699

6 MARKING

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

a) Name of the material, including the words 'Food Grade';

b) Source of manufacture;

c) Date of manufacture;

d) Minimum net contents;

e) Batch or code number; and

f) Any other requirements as specified under the *Legal Metrology (Packaged Commodities) Rules,* 2011 and *Food Safety* and *Food Safety and Standards (Packaging) Regulations, 2018 and Food Safety and Standards (Labelling and Display) Regulations, 2020.*

6.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

7 SAMPLING

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

8 TESTS

Tests shall be carried out by the methods as specified in col (4) of Table 1.

9 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) and (iii)]
METHOD OF TEST FOR DICALCIUM PHOSPHATE

A-1 PURITY

Two methods have been specified. Either could be used depending upon the facilities available.

A-1.1 METHOD I

A-1.1.1 *Reagents*

A-1.1.2 *Hydrochloric Acid Solution* **-** 10 percent (*m*/*v*).

A-1.1.3 *Methyl Orange Solution* **-** Dissolve 0.1 g of methyl orange in 100 ml of water and filter, if necessary.

A-1.1.4 *Methyl Red Solution* **-** Dissolve 0.1 ml of methyl red in 100 ml of ethanol and filter, if necessary.

A-1.1.5 *Ammonium Oxalate Solution* **-** 3.0 percent (*m*/*v*) in water.

A-1.1.6 *Ammonia Solution* **-** Dilute 400 ml of ammonium hydroxide (28 percent) with sufficient water to make 1 000 ml.

A-1.1.7 *Dilute Sulphuric Acid* **-** 1 : 6.

A-1.1.8 *Potassium Permanganate Solution* **-** 0.1 N.

A-1.2 Procedure

Weigh accurately about 0.3 g of the sample, previously dried for 3 h at 200°C. Dissolve in 10 ml of dilute hydrochloric acid solution, add about 120 ml of water and a few drops of methyl orange solution, and boil for 5 minutes, keeping the volume and *p*H of the solution in the beaker constant during the boiling period by adding hydrochloric acid or water as necessary. Add 2 drops of methyl red solution, and 30 ml of ammonium oxalate solution. Then add dropwise, with constant stirring, a mixture of equal volumes of ammonia solution and water until the pink colour of the indicator just disappears. Digest on a steam bath for 30 minutes, cool to room temperature, allow the precipitate to settle, and filter the supernatant liquid through an asbestos mat in a Gooch crucible, using gentle suction. Swirl the precipitate in the beaker with about 30 ml of a cold (below 20°C) wash solution prepared by diluting 10 ml of ammonium oxalate solution to 1 000 ml. Allow the precipitate to settle, and pour off the supernatant liquid through the filter. Repeat this washing by decantation three more times. Using the wash solution, transfer the precipitate as completely as possible to the filter. Finally, wash the beaker and the filter with two 10 ml portions of cold (below 20°C) water. Place the Gooch crucible in the beaker, and add 100 ml of water and 50 ml of cold dilute sulphuric acid. Add from a burette 35 ml of 0.1 N potassium permanganate, and stir until the colour disappears. Heat to about 70°C, and complete the titration with 0.1 N potassium permanganate. Each ml of 0.1 N potassium permanganate is equivalent to 6.803 mg of $CaHPO_3$.

A-1.2 METHOD II

A-1.2.1 *Reagents*

A-1.2.1.1 *Sodium hydroxide* **-** Dissolve 4.3 g of sodium hydroxide in water to make 100 ml.

A-1.2.1.2 *Hydroxynephthol blue indicator*

A-1.2.1.3 *Disodium ethylenediaminetetra acetate* **–** 0.05 M.

A-1.2.2 *Procedure*

Weigh accurately about 400 mg of the sample, dissolve in 150 ml of water, and add 15 ml of sodium hydroxide and 300 mg hydroxynaphthol blue indicator. Titrate with disodium ethylenediaminetetra acetate until the solution is clear. Each ml of 0.05 m dissodium ethylenediaminetetra acetate is equivalent to mg of $CaHPO_3$.

A-2 DETERMINATION OF FLUORIDE

A-2.1 Apparatus

A-2.1.1 Assembly as shown in FIG 1.

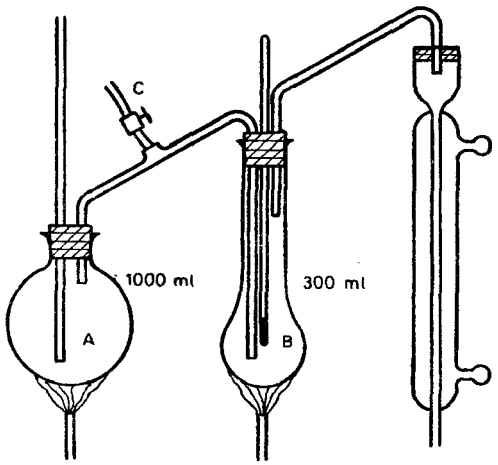

FIG 1 APPARATUS FOR LIMIT TEST FOR FLORIDE

A-2.2 Reagents

A-2.2.1 Phenolphthalein Solution - Dissolve 0.2 g of phenolphthalein in 60 ml of 90 percent ethanol and add sufficient water to make 100 ml.

A-2.2.2 Sodium Hydroxide – 0.1 N.

A-2.2.3 Sulphuric Acid – Concentrated.

A-2.2.4 Sodium Fluoride Solution – containing 50 µg of fluorine.

A-2.2.5 Hydrochloric Acid – 4 N.

A-2.2.6 Zirconium Alizarin Solution – Dissolve 0.110 g of zirconium nitrate in water, add a few drops of 4 N nitric acid, and make up to 100 ml with water. Dissolve 0.10 g of alizarin sulphonate monohydrate in 20 ml of water, and make up to 100 ml with ethanol. Mix 1 ml of first solution with one ml of the second solution and add 18 ml of water. This solution should be clear and the dilution should be prepared freshly.

A-2.3 Procedure

A-2.3.1 Place about 500 ml of water in flask A (*see* Fig 1) make it alkaline to phenolphthalein solution with N sodium hydroxide and heat the water to boiling. Leave open the screw clamp at (C).

A-2.3.2 Determine the accurate mass of the sample (M, in grams) by the formula M = 50/L, in which L is the fluoride limit, in mg/kg.

Accurately weigh the calculated amount of the sample, place it in flask 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% during the distillation. Continue until 170 ml of distillate have been collected.

A-2.3.3 Place the distillate in a l00 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 4 N hydrochloric acid and 2.0 ml of zirconium alizarin solution, and make up to 100 ml with water. Let the tubes stand for 15 minutes.

A-2.3.4 The colour of the test solution containing the sample shall not be darker than that of the standard solution.