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(पहला पुनरीक्षण)

Indian Standard

DICALCIUM PHOSPHATE, ANIMAL FEED GRADE — SPECIFICATION

(First Revision)

ICS 65.120

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

April 2002

Price Group 3

FOREWORD

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

Dicalcium phosphate is an important source of calcium and phosphorus for feeding livestock, as such or through mineral mixture. This standard, which takes into consideration the present manufacturing practices, would help in exercising proper quality control during production.

This standard prohibits the use of material of animal origin in dicalcium phosphate, as feeding of bovine origin ingredients to the ruminants, caused 'Mad cow disease' or bovine spongiform encephalopathy (BSE) in many European countries.

This standard was first published in 1969. It has been revised to include the following changes/modifications:

a) A test is incorporated to detect animal origin ingredients,

b) The requirements of Lead and Arsenic are included, and

c) The requirement of total ash is added.

In preparation of this standard considerable assistance have been received from Dr Mangat Ram Garg of *National Dairy Development Board, Anand.*

The Composition of the Committee responsible for formulation of this standard is given in Annex F.

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

DICALCIUM PHOSPHATE, ANIMAL FEED GRADE — SPECIFICATION

(First Revision)

1 SCOPE

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

2 REFERENCES

The following Indian 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:

IS No.	*Title*
460 (Part 1) : 1985	Specification for test sieves: Part 1 Wire cloth test sieves (*third revision*)
1767 : 1980	Dicalcium phosphate for dentifrice (*second revision*)
4905 : 1968	Methods for random sampling
7874	Methods of test for animal feeds and feeding stuffs:
(Part 1) : 1975	General methods
(Part 2) : 1975	Mineral and trace elements
(Part 3) : 1975	Microbiological methods
13433 (Part 1) : 1992	Animal feeds and feeding stuffs — Determination of calcium: Part 1 Titrimetric method
13574 : 1992	Animal feed and feeding stuffs — Determination of calcium and magnesium in mineral supplements
14828 : 2000	Determination of total phosphorus content — Spectrophotometric method
15121 : 2002	Animal feeding stuff — Determination of the contents of calcium, copper, iron, magnesium, manganese, potassium, sodium and zinc — Method using atomic absorption spectrometry

3 DESCRIPTION

Dicalcium phosphate, animal feed grade is odourless, white, free flowing powder.

4 REQUIREMENTS

4.1 The particle size of the material shall be such that, when sieved, not more than 10 percent of the material is retained on 200 micron IS Sieve [*see* IS 460 (Part 1)].

4.2 The material shall be free from adulterants and spores of *Bacillus anthracis, Clostridium* sp. when tested by the method described in **4.5** and **6** of IS 7874 (Part 3).

4.3 The material of animal origin shall not be used.

4.4 Dicalcium phosphate shall also conform to the requirements given in Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in moisture-proof bags, cartons, boxes or drums. All containers shall be sound, clean and free from causal agents of infectious diseases and parasites.

5.2 Marking

Each container shall be marked or labelled, giving the following particulars:

a) Name of the material,
b) Name of the manufacturer,
c) Batch or code number,
d) Net mass, and
e) Date of manufacture.

Table 1 Requirements for Dicalcium Phosphate, Animal Feed Grade

(*Clause* 4.4)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Moisture, percent by mass, *Max*	5	Annex A
ii)	Calcium (as Ca), percent by mass, *Min*	23	[1]IS 15121 or IS 13433 (Part 1) or IS 13574
iii)	Phosphorus (as P), percent by mass, *Min*	18	[2]IS 14828 or 6 of IS 7874 (Part 2)
iv)	Fluorine (as F), percent by mass, *Max*	0.10	Annex B
v)	Acid insoluble ash, percent by mass, *Max*	1.0	10 of IS 7874 (Part 2)
vi)	Lead (as Pb), mg/kg *Max*	30	A-7 of IS 1767
vii)	Arsenic (as As_2O_3), mg/kg, *Max*	10	A-6 of IS 1767
viii)	Total ash, percent by mass	73.5-78.0	Annex C
ix)	Presence of proteinous/organic impurities	Shall pass the test	Annex D

NOTE — The values for requirements ii) to viii) are on moisture-free basis.

[1]In case of dispute, the method given in IS 15121 shall be the referee method.

[2]In case of dispute, the method given in IS 14828 shall be the referee method.

5.2.1 *BIS Certification Marking*

The product may also be marked with the Standard Mark.

5.2.1.1 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.

6 SAMPLING

Representative samples of the material for testing its

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF MOISTURE

A-1 PROCEDURE

Weigh accurately about 2-3g of the sample in a silica dish. Dry the sample in vacuum oven at 60°C and 500 mm Hg pressure for 2 h.

A-1.1 Calculation

$$\text{Moisture, percent by mass} = \frac{100\,(M_1 - M_2)}{M_1 - M}$$

where

M_1 = Mass in g of the dish with the material before drying,

M_2 = Mass in g of the dish with the material after drying, and

M = Mass in g of the empty dish.

ANNEX B

[*Table* 1, *Sl No.* (iv)]

ESTIMATION OF FLUORINE BY ION SELECTIVE ELECTRODE METHOD

B-1 APPARATUS

B-1.1 Ion Selective Electrode Meter

B-1.2 Single Junction Reference Electrode

B-1.3 Solid State Fluoride Electrode

B-1.4 Magnetic Stirrer and Stir Bars

B-1.5 Plastic Labware

B-2 REAGENTS

B-2.1 Distilled or Deionized Water

B-2.2 100 mg/kg Fluoride Standard Solution

B-2.3 Reference Electrode Filling Solution

B-2.4 Total Ionic Strength Adjustment Buffer (TISAB-III)

To provide constant background ionic strength, decomplex fluoride and to adjust *pH* of the solution.

B-3 PREPARATION OF SAMPLE

Weigh accurately about 0.3 g of sample into 100 ml plastic beaker. Add 5 ml of 5 M hydrochloric acid and mix it well to dissolve all the material. Transfer the solution with deionized water. Take 25 ml aliquot in a plastic beaker and add 2.5 ml TISAB-III and measure the concentration on Ion Analyzer.

B-4 PREPARATION OF STANDARD

B-4.1 Stock Solution (500 mg/kg)

Accurately weigh 1.105 g NaF (reagent grade, dried 4 h at 100°C) into 1-litre volumetric flask. Dissolve and dilute to volume with deionized water and mix thoroughly. Store in plastic bottle at room temperature.

B-4.2 Preparation of working standard from readily available 100 mg/kg fluoride solution.

B-4.2.1 *Fluoride Standard Solution* (1 mg/kg)

Take 1 ml stock solution (100 mg/kg) into 100-ml volumetric flask and dilute to volume with deionized water and mix. It gives 1 mg/kg fluoride concentration.

B-4.2.2 *Fluoride Standard Solution* (10 mg/kg)

Take 10 ml stock solution (100 mg/kg) into 100-ml volumetric flask and dilute to volume with deionized water and mix. It gives 10 mg/kg fluoride concentration.

B-4.2.3 Take 25 ml aliquot and add 2.5 ml TISAB-III and use for meter calibration.

NOTE — Use plastic labware for fluorine estimation.

B-5 DETERMINATION OF FLUORIDE CONCENTRATION

Connect fluoride and single junction reference electrodes to ISE meter, place electrodes in standards for calibration of ISE meter. After calibration of meter with two or three standards, place electrodes in sample and stir the solution at constant rate, read concentration of standard and unknown solution directly from the meter.

B-6 CALCULATION

$$\text{Fluoride, percent by mass} = \frac{\text{Meter reading (mg/kg)} \times 100 \times 10^{-4}}{\text{Weight of sample (g)}}$$

ANNEX C

[*Table* 1, *Sl No.* (viii)]

DETERMINATION OF TOTAL ASH

C-1 PROCEDURE

Weigh accurately about 2-3 g of moisture free dicalcium phosphate (as described under Annex A) in silica dish. Complete the ignition by keeping in a muffle furnace at 550°C for 2 h. Cool in desiccator and weigh.

C-2 CALCULATION

$$\text{Total ash, percent by mass} = \frac{100\,(M_2 - M_3)}{M_1 - M}$$

where

M_2 = Mass in g of the dish with the material after drying,

M_3 = Mass in g of the dish with the material after ashing in muffle furnace,

M_1 = Mass in g of the dish with the material before drying in vacuum oven, and

M = Mass in g of the empty dish.

ANNEX D

[*Table* 1, *Sl No.* (ix)]

TEST FOR PRESENCE OF ORGANIC MATTER/PROTEINOUS IMPURITIES

Heat 0.5 g dicalcium phosphate in a dry silica dish on hot plate at 150-200°C for 30 min. Change in colour to dark grey/black with unpleasant odour indicates presence of proteinous/organic impurities.

ANNEX E

(*Clause* 6)

SAMPLING CRITERIA FOR CONFORMITY FOR DICALCIUM PHOSPHATE

E-1 GENERAL REQUIREMENTS OF SAMPLING

E-1.0 In drawing, preparing, storing and handling samples, care should be taken that the properties are not affected. The following precautions and directions shall be observed.

E-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

E-1.2 The sampling instrument shall be clean, dry and sterile when used.

E-1.3 Precautions shall be taken to protect the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

E-1.4 The samples shall be placed in clean, dry and sterile glass containers. The sample containers shall be of such a size that they are almost completely filled by the sample.

E-1.5 Each container shall be sealed air-tight with a stopper or a suitable closer after filling in such a way that it is not possible to open and reseal it without detection, and marked with full details of sampling, date of sampling, batch or code number, name of the manufacturer other important particulars of the consignment.

E-1.6 Samples shall be stored in such a manner that there is no deterioration of the material.

E-1.7 Sampling shall be done by a person agreed to between the purchaser and the vendor and if desired by any of them, in the presence of the purchaser (or his representative) and the vendor (or his representative).

E-2 SCALE OF SAMPLING

E-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 to consist of different batches of manufacture, the batches shall be grouped separately and the containers in each group shall constitute a separate lot.

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

E-2.2 The number of containers to be selected from the lot shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

Table 2 Number of Containers, to be Selected for Sampling

(*Clauses* E-2.2 *and* E-3.1)

Lot Size (*N*)	Number of Containers to be Selected (*n*)
(1)	(2)
2 to 5	2
16 to 50	3
51 to 100	4
101 to 150	5
151 to 300	7
301 and above	10

E-2.3 The containers shall be chosen at random from the lot and for this purpose a random number table as agreed to between the purchaser and the vendor shall be used (*see* IS 4905). If such a table 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 1, 2, 3 ..... etc., up to *r* and so on. Every *r*th container shall be withdrawn from the lot to give a sample for test, where $r = N/n$, *r* being the integral part of N/n; where *N* is the total number of containers in the lot, and *n* the number of containers to be selected according to Table 2. If *r* comes out to be a fractional number, its value shall be taken to be as equal to its integral part.

E-3 TEST SAMPLES AND REFEREE SAMPLE

E-3.1 Preparation of Individual Samples

Draw with an appropriate sampling instrument equal quantities of the material from different parts of each container selected according to Table 2. The total quantity of the material drawn from each container shall be not less than 1.5 kg. Mix all the portions of the material drawn from the same container thoroughly. Take out about 0.75 kg of material and divide into three equal parts. Each portion, thus obtained, shall constitute the test sample representing that particular container and shall be transferred immediately to clean and dry sample containers and sealed air-tight. These shall be labelled with particulars given under **E-1.5**. The individual samples obtained as above shall be formed into three sets in such a way that each set has a test sample representing each container selected. One of the sets shall be for the purchaser, another for the vendor and the third for the referee.

E-3.2 Preparation of Composite Sample

From the mixed material from each selected container remaining after the individual samples have been taken, equal quantities of material from each container shall be taken and mixed up together so as to form a composite sample weighing not less than 0.75 kg. This composite sample shall be divided into three equal parts and transferred to clean and dry containers and labelled with the particulars given under **E-1.5** and sealed air-tight. One of these samples shall be for the purchaser, another for the vendor and the third for the referee.

E-3.3 Referee Sample

Referee samples shall consist of a set of test samples (*see* **E-3.1**) and composite samples (*see* **E-3.2**), and shall bear the seals of the purchaser and the vendor and shall be kept at a place agreed to between the two.

E-4 TESTING OF SAMPLES

E-4.1 Samples shall be tested for each lot for ascertaining the conformity of the material to the requirements of this standard.

E-4.2 Test for calcium shall be conducted individually on each of the samples constituting the set of test samples (*see* **E-3.1**).

E-4.3 Test for the remaining characteristics, prescribed in Table 1 shall be conducted on the composite sample (*see* **E-3.2**).

E-5 CRITERIA FOR CONFORMITY

A lot shall be considered as conforming to the specification when the test results on the individual samples satisfy the requirement for calcium as specified in Table 1. The following procedure shall be adopted for determining conformity of the material for calcium.

Calculate the mean and range of the test results as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{Sum of the test results}}{\text{Number of the test samples}}$$

Range ($\bar{R}$) = Difference between the maximum and the minimum values of the test results.

If $\bar{X} - 0.4\,\bar{R}$ is greater than or equal to 23.0, the sample shall be considered as conforming to the specification in regard to calcium.

ANNEX F

(*Foreword*)

COMMITTEE COMPOSITION

Livestock Feed Sectional Committee, FAD 5

Organization	*Representative(s)*
National Dairy Development Board, Anand	DR AMRITA PATEL (***Chairman***) DR M. R. GARG (*Alternate*)
Godrej Soaps Ltd, Mumbai	DR S. L. ANAOKAR SHRI P. N. NARKHEDE (*Alternate*)
Central Institute for Research on Buffaloes, Hissar	DR T. R. CHAUHAN DR B. S. PUNIA (*Alternate*)
Mehsana District Cooperative Milk Producers Union Ltd, Mehsana	DR A. H. JOSHI DR D. P. PARMAR (*Alternate*)
Indian Veterinary Research Institute, Izatnagar	PROF N. N. PATHAK DR R. C. KATTIYAR (*Alternate*)
Punjab State Cooperative Milk Producers' Federation Ltd, Chandigarh	DR B. M. MAHAJAN SHRI A. K. DHAWAN (*Alternate*)
The Compounded Livestock Feed Manufacturing Association of India, Mumbai	CHAIRMAN
Punjab Agricultural University, Ludhiana	DR G. S. MAKKAR
Tamil Nadu Agriculture University, Chennai	DEAN
Poshak Feeds, Karnal	DR S. S. ZOMBADE
Central Sheep and Wool Research Institute, Avikanagar	DR R. C. JAKHMOLA DR S. KARIM (*Alternate*)
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Gujarat Agriculture University, Anand	DR P. M. TALPADA DR M. C. DESAI (*Alternate*)
Central Avian Research Institute, Izatnagar	DR T. S. JOHRI DR A. K. SHRIVASTAV (*Alternate*)
Central Drug Research Institute, Lucknow	SHRI P. Y. GURU
BIS Directorate General	SHRI P. K. SARKAR, Director and Head (FAD) [Representing Director General (*Ex-officio Member*)]

Member-Secretary
SHRI SUDHIR RAHI
Deputy Director (FAD), BIS

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Review of Indian Standards

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AMENDMENT NO. 1 NOVEMBER 2010
TO
IS 5470 : 2002 DICALCIUM PHOSPHATE, ANIMAL FEED GRADE — SPECIFICATION

(First Revision)

(*Page* 1, *clause* **4.1**) — Substitute the following for the existing:

'**4.1** The particle size of the material shall be such that, when sieved, not more than 10 percent of the material is retained on 212 micron IS Sieve [*see* IS 460 (Part 1)].'

[*Page* 1, *clause* **5.2**, *Sl No.* (d)] — Substitute 'Net quantity' *for* 'Net mass'.

(*Page* 1, *clause* **5.2**) — Insert the following after Sl No. (e):

'f) Any other requirements as given under the *Standards of Weights and Measures* (*Packaged Commodities*) *Rules*, 1977.'

(FAD 5)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 2 MAY 2016
TO
IS 5470 : 2002 DICALCIUM PHOSPHATE, ANIMAL FEED GRADE — SPECIFICATION

(First Revision)

(*Page* 1, *clause* **2**) — Delete 'IS 13574 : 1992 Animal feed and feeding stuffs – Determination of calcium and magnesium in mineral supplements'.

[*Page* 1, *clause* **5.2,** *Sl No*. f) (*see also Amendment No*. 1)] — Substitute '*Legal Metrology (Packaged Commodities) Rules*, 2011' *for* '*Standards of Weights and Measures (Packaged Commodities) Rules*, 1977'.

[*Page* 2, *Table* 1, *Sl No*. ii), *col* 4] — Delete 'or IS 13574'.

[*Page* 2, *Table* 1, *Sl No*. v), *col* 4] — Substitute '**10** of IS 7874 (Part 1)' *for* '**10** of IS 7874 (Part 2)'.

(*Page* 2, *clause* **6**) — Substitute the following for existing:

'**6 SAMPLING** Representative samples of the material for testing its conformity to this specification shall be drawn according to the method prescribed in Annex E'.

(*Page* 3, *clause* **B-3**, *fourth sentence*) — Substitute 'Take 25 ml aliquot in a plastic beaker and add 2.5 ml TISAB III, adjust *p*H between 5.5 and 6.0 with sodium acetate or potassium acetate and measure the concentration on ion analyzer' *for* 'Take 25 ml aliquot in a plastic beaker and add 2.5 ml TISAB-111 and measure the concentration on Ion Analyzer.'

(*Page* 4, *clause* **C-2**) — Substitute the following for the existing clause:

'**C-2 CALCULATION**

Total ash, percent by mass = $\frac{100\ (M_2\text{-}M_3)'}{M_2 - M}$

(FAD 05)

Publication Unit, BIS, New Delhi, India

AMENDMENT NO. 3 OCTOBER 2018
TO
IS 5470 : 2002 DICALCIUM PHOSPHATE, ANIMAL FEED GRADE — SPECIFICATION

(First Revision)

(*Page* 1, *clause* **4.3**) — Substitute the following for the existing clause:

'**4.3** The material of animal origin shall not be used as ingredient for ruminant feeding.'

[*Page* 2, *Table* 1, *Sl No.* iii), *col* 3] — Substitute '17.5' *for* '18'.

(FAD 05)

Publication Unit, BIS, New Delhi, India

AMENDMENT NO. 4 NOVEMBER 2020

TO

IS 5470 : 2002 DICALCIUM PHOSPHATE, ANIMAL FEED GRADE — SPECIFICATION

(First Revision)

(*Page* 2, *Table* 1, *Note*) — Substitute the following for the existing:

'NOTES:

1 The values specified for requirements given in Sl. No. (ii) to (viii) are on moisture-free basis.

2 AAS and ICP based test methods may also be used for testing the requirements. However, in case of dispute, the methods given in the Indian Standards at col 4 and wherever indicated by superscripts shall be the referee method.'

(FAD 05)

Publication Unit, BIS, New Delhi, India