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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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GB 22549-2017

Replacing GB/T 22549-2008

Feed additive - Dicalcium phosphate

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Foreword

Chapters 1, 4 and 6 of this standard are mandatory; the rest are recommended.

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard replaces GB/T 22549-2008 “Feed additive - Dicalcium phosphate”.

As compared with GB/T 22549-2008, the main technical changes except editorial modification are as follows:

- MODIFY arsenic index parameters from 0.003% to 20 mg/kg (SEE 4.2; 5.2 of 2008 version);
- DELETE dithizone spectrophotometric method in the determination of lead content (6.11.2 of 2008 version);
- ADD the chromium content index parameters and test methods (SEE 4.2, 5.13);
- ADD free water index parameters and test methods (SEE 4.2, 5.15).

This standard was proposed and shall be under the jurisdiction of the National Feed Industry Standardization Technical Committee (SAC/TC 76).

Responsible drafting organizations of this standard: CNOOC Tianjin Chemical Research Design Institute, Sichuan Feed Work Station, Sichuan Longmang Phosphorus Chemical Co., Ltd., Sinochem Yunlong Co., Ltd.

Participating drafting organizations of this standard: Yunnan Xinlong Mineral Feed Co., Ltd.

The main drafters of this standard: Li Guangming, Bai Fan, Fan Xianguo, Luo Xianming, Ma Xianlin, Yang Yinggao, Wang Yonghong.

This Standard replaces the standard previously issued as follows:

- GB/T 22549-2008.

Feed additive - Dicalcium phosphate

1 Scope

This standard specifies the requirements, test methods, inspection rules, and labeling, packaging, transportation, and storage of feed additive dicalcium phosphate.

This standard applies to the feed additive dicalcium phosphate which is produced by the wet-process phosphoric acid in accordance with a certain calcium-phosphorus ratio.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this standard.

GB/T 601 Chemical reagent - Preparations of standard volumetric solutions

GB/T 602 Chemical reagent - Preparations of standard solutions for impurity

GB/T 603 Chemical reagent - Preparations of reagent solutions for use in test methods

GB/T 6003.1-2012 Test sieves - Technical requirements and testing - Part 1: Test sieves of metal wire cloth

GB/T 6436-2002 Determination of calcium in feed

GB/T 6682-2008 Water for analytical laboratory use - Specification and test methods

GB 10648 Feed label

GB/T 13079-2006 Determination of total arsenic in feeds

GB/T 13080-2004 Determination of lead in feeds - Method using atomic absorption spectrometry

GB/T 13082-1991 Method for determination of cadmium in feeds

GB/T 13083-2002 Determination of fluorine in feed - Ion selective electrode

quimociac precipitant form precipitates. Through the filtration, drying, weighing, the total phosphorus content is calculated.

5.5.2 Reagents and materials

5.5.2.1 Hydrochloric acid solution: 1+1.

5.5.2.2 Nitric acid solution: 1+1.

5.5.2.3 Quimociac solution:

- a) WEIGH 70 g of sodium molybdate ($\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$), DISSOLVE it in 100 mL of water;
- b) WEIGH 60 g of citric acid ($\text{C}_6\text{H}_8\text{O}_7 \cdot 2\text{H}_2\text{O}$), DISSOLVE it in 150 mL of water;
- c) POUR the solution a) into solution b) whilst stirring it;
- d) ADD 25 mL of concentrated nitric acid in 100 mL of water, ADD 5 mL of quinoline;
- e) POUR solution d) into solution c). After standing for 12 hours, USE glass grit crucible, ADD 280 mL of acetone, USE water to dilute it to 1000 mL, SHAKE it uniformly, PLACE it in a polyethylene bottle for preservation.

5.5.3 Instruments

5.5.3.1 Glass grit crucible: filter plate aperture is $5\ \mu\text{m} \sim 15\ \mu\text{m}$.

5.5.3.2 Electric thermostatic oven: The temperature can be controlled at $180\ ^\circ\text{C} \pm 5\ ^\circ\text{C}$.

5.5.4 Analytical procedures

5.5.4.1 Preparation of test solution A

WEIGH about 1.0 g of specimen, accurate to 0.0002 g, PLACE in a 100 mL beaker, ADD 10 mL of hydrochloric acid and a small amount of water, COVER a watch glass, BOIL it for 10 min. After cooling, TRANSFER it into a 250 mL volumetric flask, USE water to dilute it to the mark, SHAKE it uniformly. This solution is the test solution A, which is used for the determination of total phosphorus and calcium content.

5.5.4.2 Preparation of blank test solution

Except that there is no specimen added, the reagent amount added are completely same as that for the preparation of test solution, it is subject to the same processing as that of the specimen simultaneously.

5.9.2 Reagents

Same as in chapter 4 of GB/T 13083-2002.

5.9.3 Instruments

Same as Chapter 5 of GB/T 13083-2002.

5.9.4 Analytical procedures

5.9.4.1 Preparation of test solution

WEIGH about 0.5 g ~ 1.00 g of specimen, accurate to 0.0002 g, PLACE it in a 100 mL volumetric flask, ADD 16 mL of hydrochloric acid solution (1+4), ADD water to dilute it to the mark, SHAKE it uniformly.

5.9.4.2 Determination

PIPETTE 25 mL of test solution, PLACE it in a 50 mL volumetric flask, USE total ion buffer solution to dilute it to the mark, SHAKE it uniformly. MAKE determination and calculation in accordance with chapter 7 of GB/T 13083-2002.

5.10 Determination of arsenic content

5.10.1 Method summary

Same as clause 5.1 of GB/T 13079-2006.

5.10.2 Reagents

Same as clause 5.2 of GB/T 13079-2006.

5.10.3 Instruments

Same as clause 5.3 of GB/T 13079-2006.

5.10.4 Analytical procedure

5.10.4.1 Preparation of test solution B

WEIGH 2.0 g ~ 10.0 g of specimen, accurate to 0.0002 g, ADD 20 mL of hydrochloric acid solution (1+1), HEAT to dissolve it, after cooling, TRANSFER it into a 250 mL volumetric flask, ADD water to dilute it to the mark, SHAKE it uniformly, dry FILTER it. This filtrate is test solution B, which is used for the determination of arsenic, lead, cadmium and chromium.

5.10.4.2 Determination

6.2 Sampling

Sampling in accordance with the provisions of GB/T 14699.1.

6.3 Exit-factory inspection

In the items listed in Table 1, total phosphorus, soluble phosphorus, water-soluble phosphorus, calcium, fluorine, free moisture, and fineness are the exit-factory inspection items.

6.4 Type inspection

The type inspection item is all the requirements of Chapter 4. When the product is in normal production, type inspection shall be conducted at least once every six months. However, if one of the following situations occurs, type inspection shall also be carried out:

- a) When the product is finalized;
- b) When the production process or source of raw materials has changed significantly, which may affect product quality;
- c) When the production is restored after suspension for more than three months;
- d) When there is significant difference between the exit-factory inspection result and the previous type inspection result.

6.5 Determination rules

If there is an index in the inspection result that does not conform to the requirements of this standard, the re-inspection shall be repeated on the double amount of sample which is taken from the packaging. If there is only one index that does not meet the requirements of this standard, the entire batch of products will be unqualified.

7 Labelling, packaging, transport and storage

7.1 Labels

It is performed in accordance with GB 10648.

7.2 Packaging

Packaging materials shall be non-toxic, harmless, and moisture-proof.

7.3 Transport

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