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Replacing GB/T 22548-2008

Feed additive - Monocalcium phosphate

饲料添加剂 磷酸二氢钙

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Standardization Administration of the People's Republic of China.

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Feed additive - Monocalcium phosphate

1 Scope

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

This Standard applies to the feed additive monocalcium phosphate that is produced by wet-process phosphoric acid.

Molecular formula: $Ca(H_2PO_4)_2 \cdot H_2O$

Relative molecular mass: 252.06 (according to the international relative atomic mass in 2007)

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only the dated version applies to this document. For undated references, the latest edition (including all amendments) applies to this document.

GB/T 601, Chemical reagent. Preparations of reference titration 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

water which is specified in GB/T 6682-2008. The standard titration solutions, impurity standard solutions, preparations and products that are used in the test are prepared according to the provisions of GB/T 601, GB/T 602 and GB/T 603 when no other regulations are specified.

4.3 Sensory test

The appearance is judged visually under sufficient natural light.

4.4 Identification

4.4.1 Reagents and materials

4.4.1.1 Glacial acetic acid

4.4.1.2 Hydrochloric acid solution: 1+1.

4.4.1.3 Ammonia solution: 1+1.

4.4.1.4 Ammonium oxalate solution: 100 g/L.

4.4.1.5 Silver nitrate solution: 17 g/L.

4.4.2 Identification of calcium ions

Take a small amount, about 0.1 g, of sample; add 5 mL of glacial acetic acid to dissolve; boil. Filter after cooling; add 5 mL of ammonium oxalate solution to the filtrate to give a white precipitate. This precipitate is dissolved in a hydrochloric acid solution.

4.4.3 Identification of phosphate radical

Take a small amount, about 0.1 g, of the sample; dissolve it in 10 mL of water; add 1 mL of silver nitrate solution to form a yellow precipitate. This precipitate is soluble in excess ammonia solution and insoluble in glacial acetic acid.

4.5 Determination of total phosphorus content

4.5.1 Method summary

In the acidic medium, the phosphate radical in the test solution all forms a precipitate with the added quinoxaline ketone precipitant. Calculate the content by filtration, drying, and weighing.

4.5.2 Reagents and materials

4.5.2.1 Hydrochloric acid solution: 1+1.

4.5.2.2 Nitric acid solution: 1+1.

is previously constant at 180°C ± 5°C to suction-filter the supernatant; use decantation to wash the precipitate for 5 ~ 6 times. About 20 mL of water shall be used each time; transfer the precipitate to the glass mortar; continue to use water to wash for 3 ~ 4 times. Place the glass mortar in an electrically heated drying oven; bake at 180°C ± 5°C for 45 min; take out and place in a desiccator to cool to room temperature; weigh.

4.5.5 Result calculation

The total phosphorus content is calculated, in mass fraction of phosphorus (P) w_1, according to Formula (1):

$$w_1 = \frac{(m_1 - m_0) \times 0.014\ 0}{m \times 20/250} \times 100\% \qquad \cdots\cdots(1)$$

Where:

m_1 -- the mass of quinoline phosphomolybdate precipitate that is formed by the test solution, in grams (g);

m_0 -- the mass of quinoline phosphomolybdate precipitate that is formed by the blank test solution, in grams (g);

m -- sample mass, in grams (g).

0.014 0 -- the conversion coefficient from phosphorus molybdate to phosphorus.

Take the arithmetic mean of the parallel determination results as the determination result; the absolute difference between the two parallel determination results shall not be larger than 0.2%.

4.6 Determination of water-soluble phosphorus content

Weigh approximately 0.5 g of the sample, accurate to 0.000 2 g, in a porcelain (or agate) mortar. Add water to grind; add 25 mL of water each time; continuously grind for 4 times; transfer all the aqueous solution to a 250 mL volumetric flask; shake for 30 min (2 times/s); use water to dilute to the mark; shake well. Dry filtration; discard the initial 20 mL of filtrate; pipette 20 mL of the filtrate; place it in a 250 mL beaker. The following operations are determined and calculated according to that which starts from “Add 10 mL of nitric acid solution...” of 4.5.4.3; perform a blank test at the same time.

4.7 Determination of calcium content

4.7.1 Method summary

Same as Chapter 10 of GB/T 6436-2002.

4.8.2 Reagents

Same as Chapter 4 of GB/T 13083-2002.

4.8.3 Instruments

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

4.8.4 Analysis steps

4.8.4.1 Preparation of test solution A

Weigh about 0.5 g ~ 1.00 g of sample, accurate to 0.0002 g, in a 100 mL volumetric flask; add 16 mL of hydrochloric acid solution (1+4); use water to dilute to the mark; shake well.

4.8.4.2 Determination

Pipette 25 mL of the test solution; place it in a 50 mL volumetric flask; use total ion buffer solution to dilute to the mark; shake well. Determine and calculate according to Chapter 7 of GB/T 13083-2002.

4.9 Determination of arsenic content

4.9.1 Method summary

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

4.9.2 Reagents

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

4.9.3 Instruments

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

4.9.4 Analysis steps

4.9.4.1 Preparation of test solution B

Weigh 2.0 g ~ 5.0 g of sample, accurate to 0.000 2 g; add 20 mL of hydrochloric acid solution (1+1); heat to dissolve; cool; place in a 250 mL volumetric flask; add water to dilute to the mark; shake well; dry filter. This filtrate is test solution B, which is for the determination of arsenic, lead, cadmium and chromium contents.

4.9.4.2 Determination

Take the arithmetic mean of the parallel determination results as the determination result; the absolute difference between the two parallel determination results shall not be larger than 0.5%.

4.14 Determination of fineness

4.14.1 Instruments and apparatuses

Test sieve (in accordance with GB/T 6003.1-2012): R40/3 series φ200 mm × 50 mm × 0.5 mm.

4.14.2 Analysis steps

Weigh 20.0 g of the sample, accurate to 0.01 g, on a test sieve for sieving; weigh the sieved material.

4.14.3 Result calculation

The fineness is calculated, in mass fraction w_4, according to Formula (4):

$$w_4 = \frac{m_1}{m} \times 100\% \qquad \cdots\cdots (4)$$

Where:

m_1 -- the mass of the sieved material, in grams (g);

m -- sample mass, in grams (g).

Take the arithmetic mean of the parallel determinations as the determination result. The absolute difference between the results of two parallel measurements shall not be greater than 0.3%.

4.15 pH determination

4.15.1 Instruments and apparatuses

Acidity meter: the division value is 0.02; equipped with a composite electrode or glass electrode and a calomel electrode.

4.15.2 Analysis steps

Weigh 0.24 g ± 0.01 g of the sample and place in a 150 mL beaker; add 100 mL of water to dissolve. Use the well-calibrated acidity meter to re-determine the test solution.

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