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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 1886.333-2021

**National Food Safety Standard -
Food Additives - Calcium Dihydrogen Phosphate**

食品安全国家标准 食品添加剂 磷酸二氢钙

Issued on: February 22, 2021

Implemented on: August 22, 2021

**Issued by: National Health Commission of the People's Republic of China;
State Administration for Market Regulation.**

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National Food Safety Standard - Food Additives - Calcium Dihydrogen Phosphate

1 Scope

This Standard is applicable to the food additive of calcium dihydrogen phosphate taking calcium hydrophosphate (or tricalcium phosphate, calcium hydroxide, calcium carbonate) and food additive phosphoric acid (including wet-process phosphoric acid) as raw materials.

2 Molecular Formula and Relative Molecular Mass

2.1 Molecular formula

Anhydrous calcium dihydrogen phosphate: $\text{Ca}(\text{H}_2\text{PO}_4)_2$

Calcium dihydrogen phosphate monohydrate: $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$

2.2 Relative molecular mass

Anhydrous calcium dihydrogen phosphate: 234.05 (as per 2018 internal relative atomic mass)

Calcium dihydrogen phosphate monohydrate: 252.07 (as per 2018 internal relative atomic mass)

3 Technical Requirements

3.1 Sensory requirements

The sensory requirements shall comply with the provisions of Table 1.

Table 1 – Sensory Requirements

3.2 Physicochemical index

Appendix A

Inspection Methods

Caution: Some reagents used in this test method are toxic or corrosive, so please be careful when operating! If necessary, perform it in a fume hood. If it splashes on the skin, it shall be rinsed with water immediately, and the severe cases shall be treated immediately.

A.1 General provisions

If no other requirements are specified, all the reagents and water used in this Standard refer to the analytical reagents and Class-III water specified in GB/T 6682. If no other requirements are specified, all the standard solution used in the test, standard solution for determining the impurity, preparation and product shall be prepared according to the provisions of GB/T 601, GB/T 602, GB/T 603. The solution used in the test refers to the aqueous solution when the solvent is not specified.

A.2 Identification test

A.2.1 Reagents and materials

A.2.1.1 Nitric acid solution: 1+1.

A.2.1.2 Hydrochloric acid solution: 1+3.

A.2.1.3 Ammonium oxalate solution: 35g/L.

A.2.1.4 Ammonium molybdate solution: 60g/L, take 6g of ammonium molybdate $[(\text{NH}_4)_6\text{MO}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}]$ and dissolve it in 50mL of water, slowly add 50mL of nitric acid solution while stirring, and store in a brown reagent bottle.

A.2.2 Identification method

A.2.2.1 Identification of calcium ions

Take about 0.1g of specimen; add 2mL of hydrochloric acid solution, 8mL of water; heat to 40°C ~ 50°C; add 5mL of ammonium oxalate solution; and a white precipitate is produced.

A.2.2.2 Identification of phosphate radical

Take about 0.1g of specimen; titrate nitric acid solution to dissolve the specimen; then excess 1mL of nitric acid solution; heat to 40°C ~ 50°C; add 10mL of ammonium molybdate solution; and produce yellow precipitate.

(EDTA) standard titration solution; complex with calcium ions; and use the KB mixture as an indicator to titrate the excessive ethylenediaminetetraacetic acid disodium salt (EDTA) standard titration solution by zinc sulfate standard titration solution.

A.3.2.2 Reagents and materials

A.3.2.2.1 Hydrochloric acid solution: 1+1.

A.3.2.2.2 Ammonia-ammonium chloride buffer solution (A): $\text{pH} \approx 10$.

A.3.2.2.3 Ethylenediaminetetraacetic acid disodium salt (EDTA) standard titration solution: $c(\text{EDTA}) = 0.05 \text{ mol/L}$.

A.3.2.2.4 Acid chrome blue K-naphthol green B mixed indicator liquid (KB indicator liquid).

A.3.2.2.5 Zinc sulfate standard titration solution: $c(\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}) = 0.05 \text{ mol/L}$.

Preparation: Take 15g of zinc sulfate; add water to dissolve; dilute to 1000mL with water; and shake well.

Calibration: Pipette 25.00mL of prepared zinc sulfate standard titration solution; place it in a conical flask; add 10mL of ammonia-ammonium chloride buffer solution (A) and 75mL of water; add about 0.02g of chrome black T indicator. Use 0.05mol/L ethylenediaminetetraacetic acid disodium salt (EDTA) standard titration solution to titrate until the solution turns from purple to pure blue; and it does not fade for 30s, which is the end point. At the same time, a blank test is carried out.

Except for the blank test without adding the specimen, other operations and the types and amounts of the added reagents (except the standard titration solution) are the same as the determination test.

The concentration of zinc sulfate standard titration solution c_2 , the unit is mol/L, shall be calculated according to Formula (A.2).

$$c_2 = \frac{(V_2 - V_3) \times c_3}{V_4} \dots\dots\dots (A.2)$$

Where:

V_2 - volume of ethylenediaminetetraacetic acid disodium salt (EDTA) standard titration solution consumed for titration, in mL;

V_3 - volume of ethylenediaminetetraacetic acid disodium salt (EDTA) standard titration solution consumed by blank test, in mL;

25 – volume of pipetted specimen solution, in mL;

250 – constant volume of specimen solution, in mL.

The test results are based on the arithmetic mean of the parallel determination results. The absolute difference between two independent determination results obtained under repeatability conditions is no more than 0.2%.

A.4 Determination of loss on drying

A.4.1 Apparatus

A.4.1.1 Weighing bottle: $\phi 50\text{mm} \times 30\text{mm}$.

A.4.1.2 Electric heating constant temperature drying oven: The temperature control range is $60^\circ\text{C} \pm 2^\circ\text{C}$.

A.4.2 Analysis procedures

Use a weighing bottle that has been dried at $60^\circ\text{C} \pm 2^\circ\text{C}$ for 3h; weigh about 1.5g of specimen (monohydrate), accurate to 0.0002g; and place it in an Electric heating constant temperature drying oven at $60^\circ\text{C} \pm 2^\circ\text{C}$. Dry for 3h, take out and cool to room temperature, and weigh it.

A.4.3 Calculation of results

The mass fraction w_2 of loss on drying is calculated according to Formula (A.4).

$$w_2 = \frac{m_3 - m_4}{m_5} \times 100\% \quad \dots\dots\dots (A.4)$$

Where:

m_3 – mass of specimen and weighing bottle before drying, in g;

m_4 – mass of specimen and weighing bottle after drying, in g;

m_5 – mass of specimen, in g.

The test results are based on the arithmetic mean of the parallel determination results. The absolute difference between two independent determination results obtained under repeatability conditions is no more than 0.1%.

A.5 Determination of loss on ignition

A.5.1 Apparatus

A.5.1.1 Porcelain crucible: 30mL.

Preparation of standard turbidity solution: Pipette 1.2mL of chloride standard solution; place it in a 50mL colorimetric tube; add 20mL of water, 1mL of nitric acid solution, 1mL of silver nitrate solution; dilute to the mark with water; and shake well. Place it in a dark place for 15min.

Weigh $2.00\text{g}\pm 0.01\text{g}$ of specimen; place it in a 50mL beaker; add 18mL of water and 2mL of hydrochloric acid; and heat it in a boiling water bath for 5min to dissolve. After cooling, transfer all to a 50mL colorimetric tube; dilute to the mark with water; and shake well. If the turbidity of the specimen solution is no greater than that of the standard turbidity solution, it shall be regarded as passing the test.

A.7 Determination of carbonate

A.7.1 Reagents and materials

Hydrochloric acid.

A.7.2 Analysis procedures

Weigh 2.0g of the specimen, accurate to 0.1g; place it in a 50mL conical flask; add 6mL of water to boil; titrate 2mL of hydrochloric acid after cooling. If the sample solution does not produce bubbles, it shall be regarded as passing the test.

A.8 Determination of free acid and its side salt

A.8.1 Reagents and materials

A.8.1.1 Sodium hydroxide solution: 1mol/L.

A.8.1.2 Methyl orange indicator solution: 1g/L.

A.8.2 Analysis procedures

Weigh 1.0g of specimen, accurate to 0.1g; place it in a mortar; add 3 mL of water to grind; transfer all to a 250 mL beaker. Add 100 mL of water to shake; add 1 drop of methyl orange indicator solution; and add 1 mL of sodium hydroxide solution. If the sample solution turns yellow, it shall be regarded as passing the test.

A.9 Determination of lead (Pb)

The measurement is carried out according to the method specified in Method-I of GB 5009.75-2014 or in Method-II, III, IV of GB 5009.12-2017; and the water used in the test is the Class-II water specified in GB/T 6682.

A.10 Determination of Arsenic (As)

The measurement is carried out according to the method specified in GB 5009.76 or

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