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National food safety standard - Food additives - Sodium tripolyphosphate

食品安全国家标准 食品添加剂 三聚磷酸钠

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National food safety standard - Food additives - Sodium tripolyphosphate

1 Scope

This Standard is applicable to the food additive sodium tripolyphosphate produced with sodium carbonate (or sodium hydroxide) and food additive phosphoric acid (including wet-process phosphoric acid) as raw materials, or the food additive sodium tripolyphosphate produced by recrystallization with sodium tripolyphosphate as raw material.

2 Molecular formula and relative molecular mass

2.1 Molecular formula

$Na_5P_3O_{10}$

2.2 Relative molecular mass

367.86 (according to 2018 international relative atomic mass)

3 Technical requirements

3.1 Sensory requirements

The sensory requirements shall meet the requirements of Table 1.

Table 1 -- Sensory requirements

3.2 Physical and chemical indicators

The physical and chemical indicators shall meet the requirements of Table 2.

Table 2 -- Physical and chemical indicators

Annex A

Inspection methods

WARNING: Some reagents used in this standard test method are toxic or corrosive. Be careful when operating! If splashed on the skin, rinse immediately with water. Severe cases should be treated immediately. For reagents containing highly toxic drugs, management should be strictly in accordance with relevant regulations. Avoid inhalation or contact with skin when using. It shall be carried out in a fume hood if necessary. Persons with wounds in the exposed area should not be touched. When using volatile acid, it should be carried out in a fume hood. When using flammable products, it is strictly forbidden to use an open flame for heating.

A.1 General

The reagents and water used in this Standard refer to analytically-pure reagents and grade three water specified in GB/T 6682 when other requirements are not indicated. All standard solutions, preparations and products for the determination of impurities used in the test, when no other requirements are specified, are prepared according to GB/T 601, GB/T 602, GB/T 603. The solution used refers to an aqueous solution when it is not specified which solvent is used for preparation.

A.2 Identification test

A.2.1 Reagents and materials

A.2.1.1 Hydrochloric acid.

A.2.1.2 Nitric acid solution: 1+8.

A.2.1.3 Ammonia solution: 1+1.

A.2.1.4 Sodium hydroxide solution: 40g/L.

A.2.1.5 Silver nitrate solution: 17g/L.

A.2.2 Identification methods

A.2.2.1 Identification of sodium ion

Take a platinum wire. After wetting with hydrochloric acid, burn on a colorless flame until colorless first. Re-dip the specimen. Burn in a colorless flame. The flame appears bright yellow.

phosphorus pentoxide standard solution (A.3.2.7) into a 1000mL volumetric flask. Use water to dilute to the scale mark. Mix well.

A.3.2.9 Quinomolizonone solution: Weigh 70g of sodium molybdate to dissolve in 150mL of water, as solution 1. Weigh 60g of citric acid to dissolve in a mixture of 150mL of water and 85mL of nitric acid, as solution 2. Slowly add solution 1 to solution 2 while stirring, as solution 3. In a mixture of 35mL of nitric acid and 100mL of water, add 5mL of quinoline, as solution 4. Add solution 4 to solution 3. Stir evenly. Place for 24h. Filter. Add 280mL of acetone to the filtrate. Use water to dilute to 1000mL. Mix well.

WARNING: This solution shall be stored in a polyethylene bottle. This solution contains acetone and shall not be used near the flame. Heating or boiling shall be carried out in a fume hood during operation.

A.3.2.10 Ion exchange resin: Strong alkaline anion type. Chlorine type. Particle size is 0.07mm~0.16mm. Soak in 4mol/L hydrochloric acid solution for one week. Use water to wash with the decantation method until the lotion is clear. Store in aqueous solution for later use.

A.3.2.11 Glass wool.

A.3.3 Instruments and equipment

A.3.3.1 Ion exchange column: Glass tube inner diameter is 10mm; length is 400mm. Tube bottom shrinks. Equip with a glass piston. See Figure A.1.

A.3.3.2 Liquid funnel: 125mL. It is fixed on the iron ring and connected to the top of the exchange column.

A.3.3.3 Glass sand crucible: The aperture of the filter plate is 5μm~15μm.

A.3.3.4 Hard glass test tube: ϕ25mm×200mm.

A.3.3.5 Water bath: Can be controlled at slight boiling.

A.3.3.6 Electric heating constant temperature drying oven: The temperature control range is 180°C±2°C or 250°C±5°C.

A.3.3.7 Spectrophotometer: The wavelength range is 350nm~800nm.

A.3.4 Analysis steps

A.3.4.1 Preparation of ion exchange column

Fix the ion exchange column on the shelf. Close the piston. Fill the bottom of the column with 1cm thick glass wool. Pour about 10mL of water to soak it. Pour resin into the column. Make the resin bed 30cm high. Submerge with

Before use, flow 50mL of hydrochloric acid solution through the column. Close the exchange cylinder piston. Fill the column with water. Plug the rubber stopper. Reverse several times to loosen the resin. Expel air bubbles. Fix the column vertically on the frame. Use water to slowly wash the resin first. Then at a flow rate of 5.5mL/min~6.0mL/min, wash until the pH of the effluent is 4.5~5.0 (use about 80mL of water). Maintain the liquid level 1cm above the resin layer. Close the exchange column and the piston of the separatory funnel for later use.

There shall be no air bubbles in the resin bed of the ion exchange column. After each separation, the resin must be regenerated. Keep the liquid level in the column higher than the resin layer by about 1cm during the whole process of regenerating the resin and separating the specimen. It cannot run dry.

When the resin lot number or exchange column parameters are changed, the procedure for selecting the best separation conditions shall be followed in A.3.4.4. Use a specimen of known composition. Choose a suitable elution solution. Check the accuracy of ion exchange column chromatographic separation.

A.3.4.3 Production of working curve

Accurately draw 0mL, 2mL, 4mL, 6mL, 8mL, 10mL, 15mL, 20mL, 25mL of phosphorus pentoxide standard use liquid. Transfer them into hard glass test tubes separately. Add water to dilute to 25mL. Add 10mL of ammonium molybdate-sulfuric acid solution and 2mL of ascorbic acid solution. Heat in a boiling water bath for at least 30min. Ensure complete hydrolysis. Cool to room temperature. Transfer them into 100mL volumetric flasks. Use water to dilute to the scale mark. Mix well. Use a spectrophotometer, at 650nm, water as reference and a 2cm cuvette to determine the absorbance of working curve series solution. Take the absorbance of each working curve series solution minus the absorbance of the blank solution as the abscissa, and the mass of phosphorus pentoxide (mg) as the ordinate, to draw the working curve.

A.3.4.4 Selection of the best chromatographic separation conditions

After selecting the ion exchange column, load the processed resin. Then weigh the specimen according to A.3.4.5. Prepare the specimen solution. Inject the specimen. Add the elution solution. Collect each 5mL of effluent as one portion. Transfer them into hard glass test tubes separately. Add water to dilute to 25mL. Add 10mL of ammonium molybdate-sulfuric acid solution and 2mL of ascorbic acid solution. Heat in a boiling water bath for at least 30min. Ensure complete hydrolysis. Cool room temperature. Transfer them into 100mL volumetric flasks. Use water to dilute to the scale mark. Mix well. Use a spectrophotometer, at 650nm, water as reference and a 2cm cuvette to respectively determine the absorbance. Check the corresponding phosphorus pentoxide mass (mg) from the working curve. Plot the outflow curve to determine the best separation

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 not more than 0.5%.

A.4 Determination of total phosphate (as P_2O_5)

A.4.1 Reagents and materials

A.4.1.1 Nitric acid solution: 1+1.

A.4.1.2 Quinomolimidone solution.

A.4.2 Instruments and equipment

A.4.2.1 Glass sand crucible: The aperture of the filter plate is 5μm~15μm.

A.4.2.2 Electric heating constant temperature drying oven: The temperature control range is 180°C±2°C or 250°C±5°C.

A.4.3 Analysis steps

Weigh about 1g of specimen, to the nearest of 0.0002g. Place in a 100mL beaker. Add water to dissolve. Transfer all to a 1000mL volumetric flask. Use water to dilute to the scale mark. Shake well. Filter when necessary. Pipette 25mL of test solution. Place in a 400mL tall beaker. Add 15mL of nitric acid solution and 70mL of water. Slightly boil 40min. Use water to wash the watch glass and beaker wall. The volume of the test solution is controlled to be approximately 100mL. Reheat to near boiling. Add 50mL of quinmolybdenone solution while it is hot. Cover with watch glass. Heat and boil for 1min. Keep for 30s (during the process of adding reagents and heating, do not use open flame, do not stir, so as not to condense into lumps). Cool to room temperature. Use a glass sand crucible that has been dried at 180°C±5°C for 45min to filter by decantation. Wash the precipitate 3 times in the beaker. Use 15mL of water each time. Move the precipitate into a glass sand crucible. Continue washing with water (approximately 150mL of washing water used). Put the glass sand crucible together with the precipitate in the electric heating constant temperature drying box. Start timing when the temperature stabilizes. Dry at 180°C±5°C for 45min, or dry at 250°C±5°C for 15min. Cool in a desiccator. Weigh. Conduct the blank test at the same time.

Except not adding the specimen in the blank test, the other operations and the types and amounts of reagents added are the same as those for the test specimen.

A.4.4 Result calculation

The mass fraction w_2 of total phosphate (calculated as P_2O_5) is calculated according to formula (A.2).

$$w_3 = \frac{m_6 - m_7}{m_8} \times 100\% \quad \cdots\cdots (A.3)$$

Where,

m_6 - The mass of water insoluble matter and glass sand crucible, in grams (g);

m_7 - The mass of glass sand crucible, in grams (g);

m_8 - The mass of specimen, in grams (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 not more than 0.01%.

A.6 Determination of pH (10g/L aqueous solution)

A.6.1 Reagents and materials

Carbon dioxide-free water.

A.6.2 Instruments and equipment

Acidity meter: Resolution is 0.01pH; equipped with glass electrode and saturated calomel electrode (or composite electrode).

A.6.3 Analysis steps

Weigh 1.00g±0.01g of specimen. Place in a 100mL beaker. Use carbon dioxide-free water to dissolve. Transfer to a 100mL volumetric flask. Use carbon dioxide-free water to dilute to the scale of mark. Shake well. Pour into a 100mL dry beaker. Use a calibrated acid meter to measure the pH of the specimen solution.

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 not more than 0.1.

A.7 Determination of lead

Measure according to the method specified in GB 5009.75 or GB 5009.12. The water used in the test is the grade two water specified in GB/T 6682.

A.8 Determination of arsenic (As)

Measure according to the method specified in GB 5009.76 or GB 5009.11. The water used in the test is the grade two water specified in GB/T 6682.

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