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National food safety standard - Food additives - Sodium pyrophosphate

食品安全国家标准 食品添加剂 焦磷酸钠

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1 Scope

This Standard applies to food additive sodium pyrophosphate that is produced with sodium carbonate (or sodium hydroxide) and the food additive phosphoric acid (including wet-process phosphoric acid) as raw materials.

2 Molecular formula and relative molecular mass

2.1 Molecular formula

Anhydrous sodium pyrophosphate: $Na_4P_2O_7$

Sodium pyrophosphate decahydrate: $Na_4P_2O_7 \cdot 10H_2O$

2.2 Relative molecular mass

Anhydrous sodium pyrophosphate: 265.91 (according to 2018 international relative atomic mass)

Sodium pyrophosphate decahydrate: 446.07 (according to 2018 international relative atomic mass)

3 Technical requirements

3.1 Sensory requirements

Sensory requirements shall be in accordance with Table 1.

Table 1 – Sensory requirements

3.2 Physical and chemical indicators

Physical and chemical indicators shall be in accordance with Table 2.

Table 2 – Physical and chemical indicators

Appendix A

Inspection method

WARNING: Some reagents which are used in the test method of this Standard are toxic or corrosive; be careful during the operation. If it splashes on the skin or eyes, use plenty of water to rinse immediately; if it is serious, seek medical attention immediately.

A.1 General provisions

The reagents and water that are used in this Standard, when no other requirements are specified, refer to analytical reagents and grade-III water which is specified in GB/T 6682. The standard titration solutions, preparations and products, which are used in the test, are all prepared in accordance with GB/T 601, GB/T 602, and GB/T 603, unless other requirements are specified. The used solution, if not indicated which solvent is used, refers to aqueous solution.

A.2 Identification test

A.2.1 Reagents and materials

A.2.1.1 Hydrochloric acid.

A.2.1.2 Nitric acid solution: 1+9.

A.2.1.3 Quinomolimidone solution: Prepare according to the requirements of HG/T 3696.3.

A.2.1.4 Platinum wire ring.

A.2.2 Identification method

A.2.2.1 Sodium ion

Weigh 1 g of the sample; add 20 mL of water to dissolve; use hydrochloric acid to wet the platinum wire ring; then, burn it on a colorless flame until it is colorless; then, dip the test solution and burn it in a colorless flame; the flame shall be bright yellow.

A.2.2.2 Pyrophosphate ion

A.2.2.2.1 Sample solution: Dissolve 0.1 g of the sample in 100 mL of nitric acid solution.

Third crystallization: Recrystallize the second crystallization according to the method of the second crystallization.

b) Preparation method with reagent sodium pyrophosphate decahydrate as raw material:

Weigh 80 g of the reagent sodium pyrophosphate decahydrate; operate according to the first and second crystallization methods in a).

Place the sodium pyrophosphate that is recrystallized by the above method in a porcelain crucible; burn at 400° C to a constant mass.

A.3.2.5 Sodium hydroxide standard titration solution: c(NaOH) = 0.1 mol/L.

Calibration: Weigh about 0.5 g of anhydrous sodium pyrophosphate (A.3.2.4), accurate to 0.000 2 g; place it in a 250 mL beaker; add 90 mL of water to dissolve; add hydrochloric acid solutions (A.3.2.1, A.3.2.2) to adjust the pH of the solution to 3.8. Add 50 mL of zinc sulfate solution; stir for 5 min; use sodium hydroxide standard titration solution to titrate under stirring, until the pH of the solution is close to 3.6; stir for 2 min to make the solution reach equilibrium; continue to titrate until the pH is 3.8; stir for 30 seconds after each drop at this time.

The number of grams ρ of 0.1 mol/L sodium hydroxide standard titration solution per milliliter that is equivalent to sodium pyrophosphate, in grams per milliliter (g/mL), is calculated according to Formula (A.1).

$$\rho = \frac{m_1}{V_1} \qquad \text{……(A.1)}$$

Where:

m_1 – mass of the weighted anhydrous sodium pyrophosphate, in grams (g);

V_1 – volume of the sodium hydroxide standard titration solution that is consumed in calibration, in milliliters (mL).

A.3.3 Instruments and apparatuses

A.3.3.1 Potentiometric titrator or pH meter: the resolution is 0.01 mV or 0.02 pH.

A.3.3.2 Electromagnetic stirrer.

A.3.4 Analysis steps

Weigh about 5 g of the sample, accurate to 0.000 2 g; dissolve the sample in water; transfer to a 500 mL volumetric flask; dilute to the mark and shake well; filter if necessary.

A.4.1.3 Porcelain crucible.

A.4.2 Analysis steps

Weigh about 5 g of the sample, accurate to 0.000 2 g; place it in a porcelain crucible that has been burn to a constant mass at 800 °C ± 25 °C; bake it at 110 °C ± 2 °C for 4 h; then, move it into a high-temperature furnace at 800 °C ± 25 °C to burn for 30 minutes; cool to room temperature in a desiccator; weigh.

A.4.3 Result calculation

Calculate the mass fraction w_3 of loss on ignition according to Formula (A.4).

$$w_3 = \frac{m_3 - m_4}{m_5} \times 100\% \qquad \cdots\cdots (A.4)$$

Where:

m_3 – mass of the sample and the porcelain crucible before firing, in grams (g);

m_4 – mass of the sample and the porcelain crucible after firing, in grams (g);

m_5 – sample mass, in grams (g).

The test result is based on the arithmetic mean of the parallel determination results. The absolute difference between two independent determination results that are obtained under repeatability conditions is: not more than 0.1% for sodium pyrophosphate decahydrate; not more than 0.02% for anhydrous sodium pyrophosphate.

A.5 Determination of water insoluble matter

A.5.1 Instruments and apparatuses

A.5.1.1 Glass sand crucible: The aperture of the filter plate is 5 μm ~ 15 μm.

A.5.1.2 Electrothermal constant-temperature dry box: The temperature control range is 105 °C ± 2 °C.

A.5.2 Analysis steps

Weigh about 20 g of the sample, accurate to 0.01 g; place it in a 400 mL beaker; add 200 mL of water; heat to dissolve it. While it is hot, use a glass sand crucible of constant mass at 105 °C ± 2 °C to filter; use hot water to wash it until the filtrate is alkali-free. Place the glass sand crucible and water-insoluble matter in an electrothermal constant-temperature dry box at 105 °C ± 2 °C to dry until the mass is constant.

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