



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB 1886.4-2020

**National food safety standard - Food additives - Sodium
Hexametaphosphate**

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1 Scope

This Standard is applicable to food additive sodium hexametaphosphate, which is generated by the melt polymerization reaction of sodium carbonate or sodium hydroxide and food additive phosphoric acid (including phosphoric acid by wet process).

2.1 Sensory Requirements

Sensory requirements shall comply with the stipulations of Table 1.

Table 1 -- Sensory Requirements

Item	Requirements	Inspection Methods
Color	Colorless and transparent, or white	Take an appropriate amount of specimen; place it in a 50 mL beaker; under natural light, observe its color and state
State	Glassy flake, granule or powder	

2.2 Physical and Chemical Indicators

The physical and chemical indicators shall comply with the stipulations of Table 2.

Appendix A Inspection Methods

A.1 Warning Some reagents used in the inspection methods of this Standard are toxic or corrosive, so be careful when handling them! If they splash on the skin or eyes, immediately use plenty of water for rinsing. In severe cases, medical treatment shall be immediately sought. For the flammable products used in the tests, no open flame shall be used during operation.

A.2 General Rules Unless other requirements are indicated, the reagents and water used in this Standard refer to analytically pure reagents and Grade-3 water specified in GB/T 6682. Unless other requirements are indicated, the standard titration solutions, preparations and products used in the tests are all prepared in accordance with GB/T 601, GB/T 602 and GB/T 603. When it is not specified which solvent is used for preparation, the used solution refers to an aqueous solution.

A.3 Identification and Test A.3.1 Reagents and materials A.3.1.1 Hydrochloric acid.

A.3.1.2 Nitric acid solution: 1 + 7.

A.3.1.3 Silver nitrate solution: 17 g/L.

A.3.1.4 Sodium hydroxide solution: 40 g/L.

A.3.1.5 Platinum-wire ring.

A.3.2 Identification methods A.3.2.1 Sodium ion After using hydrochloric acid to wet the platinum-wire ring, burn it on colorless flame, until it becomes colorless. Dip the specimen; burn it in colorless flame; the flame shall be luminous yellow.

A.3.2.2 Phosphate Weigh-take 0.1 g of specimen; place it in a 50 mL beaker. Add 5 mL of hot nitric acid A.5.3.1 Sintered-glass filter crucible: the aperture of the filter plate is 5 μm ~ 15 μm .

A.5.3.2 Electrothermal constant-temperature drying oven: the temperature is controlled at 180 $^{\circ}\text{C}$ $\pm$ 5 $^{\circ}\text{C}$ or 250 $^{\circ}\text{C}$ $\pm$ 10 $^{\circ}\text{C}$.

A.5.4 Analytical procedures Use a pipette to transfer-take 50 mL of the specimen solution (see A.4.4.1); place it in a 100 mL volumetric flask. While constantly shaking it, add 30 mL of barium chloride solution; thoroughly shake it to complete the precipitation. Then, use water to dilute to the scale; shake it well; dry-filter it. Use a pipette to transfer-take 50 mL of filtrate; place it in a 400 mL tall beaker. Add 15 mL of nitric acid solution and 35 mL of water; heat it up and boil it for 15 min. While it is still hot, add 20 mL of quin-molybdenum-ketone solution; use a watch glass to cover it; preserve the heat for 10 min (in the process of adding reagents and heating, DO NOT use open flame, DO NOT stir it, so as avoid condensation into lumps), then, cool it down to room temperature. Use a sintered-glass filter crucible, which is dried to a constant mass at 180 $^{\circ}\text{C}$ $\pm$ 5 $^{\circ}\text{C}$ or 250 $^{\circ}\text{C}$ $\pm$ 10 $^{\circ}\text{C}$, to filter it by the pouring method. In a beaker, rinse and precipitate it for 3 times, with 15 mL of water each time. Transfer all the precipitate to the sintered-glass filter crucible, then, continue to use water to rinse it (the used rinsing water is about 150 mL). At 180 $^{\circ}\text{C}$ $\pm$ 5 $^{\circ}\text{C}$, dry it for 45 min, or at 250 $^{\circ}\text{C}$ $\pm$ 10 $^{\circ}\text{C}$, dry it for 30 min. In a desiccator, cool it down; weigh it.

A.5.5 Result calculation The mass fraction w_2 of inactive phosphate (calculated as P_2O_5) shall be calculated in accordance with Formula (A.2):

Where, m_3 ---the mass of quinoline phosphomolybdate precipitate, expressed in (g);

0.03207---conversion factor of quinoline phosphomolybdate and phosphorus pentoxide;

20---dilution factor;

m_2 ---the mass of specimen (see A.4.4.1), expressed in (g).

The test results shall be subject to the arithmetic mean value of the parallel determination

results. The absolute difference between two independent determination results obtained under repeatability conditions is not greater than 0.3%.

A.6 pH (10 g/L solution) Determination The test results shall be subject to the arithmetic mean value of the parallel determination results. The absolute difference between two independent determination results obtained under repeatability conditions is not greater than 0.1%.

A.8 Determination of Iron (Fe)

A.8.1 Method summary Use ascorbic acid to reduce the trivalent iron ions in the specimen solution to divalent iron ions. When the pH is 2 ~ 9, the divalent iron ions and o-phenanthroline generate orange-red complexes. Use a spectrophotometer to determine its absorbance at the wavelength of 510 nm.

A.8.2 Reagents and materials Same as Chapter 4 of GB/T 3049-2006.

A.8.3 Instruments and equipment Same as Chapter 5 of GB/T 3049-2006.

A.8.4 Analytical procedures A.8.4.1 Drawing of standard curve In accordance with the stipulations of 6.3 in GB/T 3049-2006, in six 100 mL volumetric flasks, prepare series standard solutions with the mass (mg) of iron (Fe) per 100 mL:

0.00 mg, 0.01 mg, 0.02 mg, 0.04 mg, 0.08 mg and 0.10 mg. Use 4 cm or 5 cm cuvette to determine the absorbance of the standard solutions. Take the mass (mg) of iron (Fe) per 100 mL as the x-coordinate; take the corresponding absorbance as the y-coordinate; draw a standard curve.

A.8.4.2 Determination Weigh-take about 3 g of specimen, accurate to 0.0002 g; place it in a 250 mL beaker.

Add 50 mL of water and 10 mL of hydrochloric acid solution; use a watch glass to cover it. Heat it up and boil it for 15 min, then, cool it down to room temperature. Transfer all to a 250 mL volumetric flask; use water to dilute to the scale; shake it well. Use a pipette to transfer-take 25 mL of the test solution; place it in a 100 mL volumetric flask.

The subsequent steps shall follow the operations in 6.4 of GB/T 3049-2006, starting from "if necessary, add water to 60 mL.....".

At the same time, conduct a blank test. Except that no specimen is added, the blank test solution is the same as the test solution in the other operations and the added reagents.

In accordance with the measured absorbance, from the standard curve, find out the electrode as the measuring electrode; use the standard curve method to determine the fluoride content.

A.12.2 Reagents and materials A.12.2.1 Hydrochloric acid solution: 1 + 4.

A.12.2.2 Total ionic strength adjustment buffer (TISAB): in accordance with the requirements of HG/T 3696.3, prepare it right before use.

A.12.2.3 Fluoride (F) standard solution: 1 mL contains 0.01 mg of fluoride (F). Use a pipette to transfer-take 10 mL of fluoride (F) standard solution, which is prepared in accordance with GB/T 602; place it in a 100 mL volumetric flask; use water to dilute to the scale; shake it well.

A.12.3 Instruments and equipment A.12.3.1 pH meter: with a division value of 1 mV; equipped with saturated calomel electrode and fluoride ion selective electrode.

A.12.3.2 Magnetic stirrer.

A.12.4 Analytical procedures A.12.4.1 Drawing of standard curve In five 50 mL volumetric flasks, use a pipette to respectively add 1.00 mL, 2.00 mL,

3.00 mL, 4.00 mL and 5.00 mL of the fluoride (F) standard solution; accurately add 4

mL of hydrochloric acid solution; add 25 mL of the total ionic strength adjustment buffer (TISAB); use water to dilute to the scale; shake it well. Connect the fluoride ion selective electrode and the saturated calomel electrode to the negative and positive ends of the pH meter. Insert the electrode into a 50 mL polyethylene beaker, which is filled with water. Under electromagnetic stirring, read the equilibrium potential value, replace the water, in which, the electrode is immersed, then, after the potential value reaches an equilibrium, from low to high concentration, respectively determine the potential value of the fluorine standard solution. Take the potential value as the y-coordinate; take the logarithm of the fluoride mass (mg) as the x-coordinate; draw a standard curve.

A.12.4.2 Determination Weigh-take about 1 g of specimen, accurate to 0.0002 g; place it in a 50 mL polyethylene beaker. Add a small amount of water to moisten it; accurately add 4 mL of hydrochloric acid solution to dissolve it. Then, transfer all to a 50 mL volumetric flask.

Add 25 mL of the total ionic strength adjustment buffer (TISAB); use water to dilute to the scale; shake it well. Determine the potential value of the test solution. In accordance with the determined potential value, from the standard curve, find out the