GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

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National Food Safety Standard

- Food Additives - Phosphoric Acid

食品安全国家标准 食品添加剂 磷酸

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National Food Safety Standard

– Food Additives – Phosphoric Acid

1 Scope

This Standard is applicable to food additive of phosphoric acid produced by thermal method.

2 Molecular Formula and Relative Molecular Mass

2.1 Molecular formula

H_3PO_4

2.2 Relative molecular mass

97.99 (according to international relative atomic mass in 2007)

3 Technical Requirements

3.1 Sensory requirements

The sensory requirements shall meet the requirements of Table 1.

3.2 Physical and chemical indicators

The physical and chemical indicators shall conform to the provisions of Table 2.

weighing, the phosphoric acid content was determined.

A.4.1.2 Reagents and materials

A.4.1.2.1 Hydrochloric acid.

A.4.1.2.2 Preparation of quimociac solution

a) Take 70g of sodium molybdate to dissolve in 150mL of water; this solution is Solution-A;

b) Take 60g of citric acid to dissolve in a mixed solution of 150mL of water and 85mL of nitric acid; this solution is Solution-B;

c) Pour Solution-A into Solution-B under stirring condition; this solution is Solution-C;

d) Add 35 mL of nitric acid to 100 mL of water, and then add 5 mL of quinoline; this solution is Solution-D;

e) Pour Solution-D into Solution-C and mix well. After standing for 12h, filter by a glass sand crucible; add 280 mL of acetone; dilute with water to 1000 mL and mix well; and store in a polyethylene bottle.

A.4.1.3 instruments and equipment

A.4.1.3.1 Glass sand crucible: filter plate pore size 5μm~15μm.

A.4.1.3.2 Electric oven: the temperature can be controlled at 180°C±5°C or 250°C±10°C.

A.4.1.4 Analysis procedures

A.4.1.4.1 Preparation of test solution

Take about 1g of specimen, accurate to 0.0002g; put it in a 100mL beaker; add 5mL of hydrochloric acid and an appropriate amount of water; cover the watch glass; boil for 10min; after cooling, transfer to a 500mL volumetric flask; add 10mL of hydrochloric acid, and dilute with water to the mark, shake well.

A.4.1.4.2 Preparation of blank test solution

While preparing the test solution, except for not adding specimen, other operations and the added amounts of reagents are the same as those in A.4.1.4.1.

A.4.1.4.3 Determination

Pipette 10mL of test solution and blank test solution into 250mL beakers respectively;

A.5.2 Reagents and materials

A.5.2.1 Hydrochloric acid solution: 1+1.

A.5.2.2 Nitric acid solution: 1+15.

A.5.2.3 Sodium hydroxide solution: 200 g/L.

A.5.2.4 Citric acid - trisodium citrate buffer solution: pH = 5.5~6. Take 270g of trisodium citrate ($Na_3C_6H_5O_7 \cdot 2H_2O$) and 24g of citric acid ($C_6H_6O_7 \cdot H_2O$) to dissolve in water, dilute to 1000mL, and mix well.

A.5.2.5 Bromocresol green indicator liquid: 1 g/L.

A.5.2.6 Fluoride standard solution: 1mL of such solution contains 2μg of fluorine (F). Prepare just before use. Pipette 2mL of the fluoride standard solution prepared according to GB/T 602; place it in a 100mL volumetric flask; dilute with water to the mark; and shake well.

A.5.3 Instruments and equipment

A.5.3.1 Fluoride ion selective electrode.

A.5.3.2 Saturated calomel electrode.

A.5.3.3 Potentiometer: The accuracy is 2mV/div.

A.5.3.4 Electromagnetic stirrer.

A.5.4 Analysis procedures

A.5.4.1 Drawing of working curve

Pipette 0.00mL, 1.00mL, 3.00mL, 5.00mL, 7.00mL, 10.00mL of fluoride standard solutions into the 50mL volumetric flasks, respectively; add 1mL hydrochloric acid solution, 5 drops of citric acid - trisodium citrate buffer solution and 2 drops of bromocresol green indicator liquid; adjust the solution to blue by sodium hydroxide solution; then adjust the solution to just yellow by nitric acid solution; add 20mL of citric acid - trisodium citrate buffer solution; and dilute with water to scale and mix well. Pour the solution into a 50mL dry beaker; place it on an electromagnetic stirrer; insert a fluoride ion selective electrode and a saturated calomel electrode; connect the potentiometer; stir for a while; adjust the potentiometer to zero point; then measure; and record the potential at equilibrium value. Draw the standard curve with the logarithmic value of the value of the mass of fluoride (by F) as the abscissa and the corresponding potential value as the ordinate.

A.5.4.2 Preparation of test solution

In cerium sulfate standard titration solution with excessive acid, it reacts with the readable oxidizable substance in the specimen. Titrate the excessive cerium sulfate standard titration solution by ammonium ferrous sulfate standard titration solution.

A.6.2 Reagents and materials

A.6.2.1 Sulfuric acid solution: 1+2.

A.6.2.2 Silver sulfate solution (10 g/L): take 1g of silver sulfate; add 50mL of sulfuric acid solution (1+2) to dissolve; dilute with water to 100mL; and shake well.

A.6.2.3 Cerium sulfate standard titration solution: $c[Ce(SO_4)_2] = 0.1$ mol/L.

A.6.2.4 Ammonium ferrous sulfate Standard titration solution: $c[(NH_4)_2Fe(SO_4)_2] = 0.1$ mol/L.

A.6.2.5 1,10-phenanthroline-ferrous iron indicator liquid.

A.6.3 Analysis procedures

In a 250mL conical flask, add 40mL of sulfuric acid solution; use a pipette to transfer 10.00mL of cerium sulfate standard titration solution; and then add 10mL of silver sulfate solution; shake well. Add 10g (about 6mL) of the specimen dropwise; add 40 mL of water; boil for 15 min, and cool. Dilute with water to the original volume; add 2 drops of 1,10-phenanthroline-ferrous iron indicator liquid; and titrate by ammonium ferrous sulfate standard titration solution until the solution turns red. Do a blank test at the same time.

A.6.4 Calculation of results

Mass fraction, w_3, of readily oxidizable substance (by H_3PO_3) is calculated according to Formula (A.4):

$$w_3 = \frac{\frac{V_1 - V_2}{1\,000} \times c \times M}{m} \times 100\% \qquad \cdots\cdots (A.4)$$

Where:

V_1 – volume of ammonium ferrous sulfate standard titration solution (A.6.2.4) consumed in the blank test, in mL;

V_2 – volume of ammonium ferrous sulfate standard titration solution (A.6.2.4) consumed during the titration, in mL;

1000 – conversion coefficient between mL and L;

corresponding absorbance as the ordinate to draw a working curve.

A.7.1.4.2 Determination

Take about 10g of specimen, accurate to 0.01g; put it in the arsenic generation bottle; add water to 40mL; and mix well. Add 20mL of sulfuric acid solution and mix well. Add 2mL of potassium iodide solution and 2mL of stannous chloride solution; and shake well. Stand for 15min. Add 3mL of silver diethyldithiocarbamate-pyridine solution to the absorption tube containing lead acetate cotton; add 3g of arsenic-free metal zinc particles to the arsenic generation bottle; and immediately connect the absorption tube to the arsenic generation bottle. After the reaction is complete (approximately 45min at normal temperature), the absorbance of the absorption solution in the absorption tube is measured at 540nm by a 1cm colorimetric cell. Subtract the absorbance of the blank solution from the absorbance of the test solution, and find the corresponding arsenic (As) content from the working curve.

A.7.1.5 Calculation of results

Mass fraction, w_4, of arsenic (As) in milligrams per kilogram (mg/kg) is calculated according to Formula (A.5):

$$w_4 = \frac{m_1}{m} \times 100\% \qquad \cdots\cdots (A.5)$$

Where:

m_1 – mass fraction of arsenic found from the working curve, in μg;

m – specimen mass, in g.

Take the arithmetic mean of the parallel determination results as the determination result; the absolute difference of the parallel determination shall be no greater than 0.2 mg/kg.

A.7.2 Arsenic spot method

A.7.2.1 Method summary

In sulfuric acid medium, arsenic is reduced to hydrogen arsenide by metal zinc. Hydrogen arsenide reacts with mercury bromide to form red-brown or light-yellow arsenic spots, which are compared with standard spots.

A.7.2.2 Reagents and materials

A.7.2.2.1 Arsenic-free zinc particles.

A.7.2.2.2 Sulfuric acid solution: 1+4.

sulfuric acid solution, and mix well. Add 2mL of potassium iodide solution and 2mL of stannous chloride solution; and shake well. Stand for 15min. Put the mercury bromide test paper into the test tube containing lead acetate cotton as shown in Figure A.2; add 3g of arsenic-free zinc particles to the arsenic generation bottle; and immediately connect the test tube to the arsenic generation bottle. After the reaction is complete (approximately 45min at normal temperature), take out the mercury bromide test paper and compare it with the standard spots. The red-brown arsenic spot shall be no deeper than the standard spots.

A.8 Determination of heavy metals (by Pb)

A.8.1 Method summary

Under the condition of pH 3~4, the heavy metal ions in the specimen interact with hydrogen sulfide to form a brown suspension, which is compared with the lead standard solution treated in the same way.

A.8.2 Reagents and materials

A.8.2.1 Glacial acetic acid.

A.8.2.2 Ammonia solution: 1+1.

A.8.2.3 Preparation of sodium sulfide-glycerol solution: take 5g of sodium sulfide and dissolve it in 10mL of water and 30mL of glycerol mixed solution. This solution shall be stored in a brown bottle, protected from light, and sealed for three months.

A.8.2.4 Lead standard solution: 1mL of such solution contains 10μg of lead (Pb); prepare just before use. Pipette 10 mL of lead standard solution prepared according to GB/T 602; place it in a 100mL volumetric flask; dilute to the mark with water; and shake well.

A.8.2.5 Preparation of lead standard colorimetric solution: pipette 5mL of test solution and appropriate amount (1mL ~ 3mL) of lead standard solution; place them in 50mL colorimetric tube; and treat with the test solution in the same way and at the same time. In order to obtain the content of heavy metals, a series of standard lead colorimetric solutions need to be prepared.

A.8.3 Analysis procedures

A.8.3.1 Preparation of test solution

Take about 10g of specimen, accurate to 0.01g. Place in a 50mL volumetric flask; dilute to the mark with water; and shake well.

A.8.3.2 Determination

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