GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

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National Food Safety Standard - Food Additive - Phosphoric Acid (wet process)

食品安全国家标准

食品添加剂 磷酸（湿法）

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National Food Safety Standard - Food Additive - Phosphoric Acid (wet process)

1 Scope

This Standard is applicable to food additive - phosphoric acid (wet method) obtained by the purification process of wet-process crude phosphoric acid through impurity removal, solvent extraction and refining, etc.

2 Molecular Formula and Relative Molecular Mass

2.1 Molecular Formula

H_3PO_4

2.2 Relative Molecular Mass

97.99 (in accordance with the international relative molecular mass of Year 2016)

3 Technical Requirements

3.1 Sensory Requirements

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

Table 1 -- Sensory Requirements

3.2 Physical and Chemical Indicators

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

A.4.1.1 Method summary

The sample is under acidic conditions. Purge with nitrogen to remove inorganic carbon. Through sodium sulfate oxidation, it is quickly generated into carbon dioxide at 100 °C. Through an infrared analyzer, the content of organic carbon is detected.

A.4.1.2 Reagents and materials

A.4.1.2.1 Sodium persulfate solution: 100 g/L. Weigh-take 100 g of sodium persulfate, dissolve it in an appropriate amount of carbon dioxide-free water; use carbon dioxide-free water to dilute it to 1,000 mL.

A.4.1.2.2 Phosphoric acid solution: 5%. Measure-take 59 mL of phosphoric acid; use carbon dioxide-free water to dilute it to 1,000 mL.

A.4.1.2.3 Total carbon standard stock solution: 1 mL of solution contains 1.0 mg of carbon (C). Weigh-take 2.1254 g of reference potassium hydrogen phthalate dried at 120 °C ± 2 °C for 2 h. Add carbon dioxide-free water to dissolve it, then, transfer it into a 1,000 mL volumetric flask. Use carbon dioxide-free water to dilute to the scale, then, shake it well.

A.4.1.2.4 Total carbon standard service solution: 1 mL of solution contains 0.1 mg of carbon (C). Use a pipette to transfer-take 10 mL of total carbon standard stock solution (see A.4.1.2.3); place it in a 100 mL volumetric flask. Use carbon dioxide-free water to dilute it to the scale, then, shake it well.

A.4.1.2.5 Carbon dioxide-free water: prepare it in accordance with the method specified in GB/T 603.

A.4.1.3 Instruments and equipment

Total organic carbon (TOC) analyzer: the used high-purity nitrogen complies with the requirements of GB/T 8979.

A.4.1.4 Analytical procedures

A.4.1.4.1 Drawing of standard curve

Respectively transfer-take 0.00 mL, 1.00 mL, 2.00 mL, 3.00 mL and 4.00 mL of the total carbon standard service solution; respectively place them in a 100 mL volumetric flask. Use carbon dioxide-free water to dilute to the scale, then, shake it well. Adjust the total organic carbon (TOC) analyzer to the optimal test conditions. In accordance with the sequence of concentration from low to high, successively conduct the determination. For each standard solution being determined, the instrument automatically inhales 5.00 mL of corresponding standard solution, 0.50 mL of phosphoric acid solution and 1.00 mL of sodium persulfate solution. After the reaction is completed, the instrument automatically measures the peak area of the standard

carbon (IC).

A.4.2.2 Reagents and materials

A.4.2.2.1 Total carbon standard stock solution: 1 mL of solution contains 1.0 mg of carbon (C). Weigh-take 2.1254 g of reference potassium hydrogen phthalate dried at 120 °C ± 2 °C for 2 h. Add carbon dioxide-free water to dissolve it, then, transfer it into a 1,000 mL volumetric flask. Use carbon dioxide-free water to dilute to the scale, then, shake it well.

A.4.2.2.2 Total carbon standard service solution: 1 mL of solution contains 0.1 mg of carbon (C). Use a pipette to transfer-take 10 mL of total carbon standard stock solution (see A.4.2.2.1), place it in a 100 mL volumetric flask. Use carbon dioxide-free water to dilute to the scale, then, shake it well.

A.4.2.2.3 Inorganic carbon standard stock solution: 1 mL of solution contains 1.0 mg of carbon (C). Weigh-take 3.50 g of sodium bicarbonate previously dried in a silica gel drier for 2 h, and 4.41 g of reference sodium carbonate dried at 280 °C ~ 290 °C for 1 h. Add carbon dioxide-free water to dissolve it, then, transfer it into a 1,000 mL volumetric flask. Use carbon dioxide-free water to dilute to the scale, then, shake it well.

A.4.2.2.4 Inorganic carbon standard service solution: 1 mL of solution contains 0.01 mg of carbon (C). Use a pipette to transfer-take 1.00 mL of the inorganic carbon standard stock solution (see A.4.2.2.3); place it in a 100 mL volumetric flask. Use carbon dioxide-free water to dilute to the scale, then, shake it well.

A.4.2.2.5 Carbon dioxide-free water: prepare it in accordance with the method specified in GB/T 603.

A.4.2.3 Instruments and equipment

Total organic carbon (TOC) analyzer: the used high-purity oxygen complies with the requirements of GB/T 14599.

A.4.2.4 Analytical procedures

A.4.2.4.1 Drawing of standard curve

Respectively transfer-take 0.00 mL, 1.00 mL, 2.00 mL, 3.00 mL and 4.00 mL of the total carbon standard service solution; respectively place them in a 100 mL volumetric flask. Use carbon dioxide-free water to dilute to the scale, then, shake it well. Adjust the total organic carbon (TOC) analyzer to the optimal test conditions. Draw the standard solution to measure the peak area. Subtract the peak area of the blank solution from the peak area of each standard solution. Take the mass concentration (mg/L) of carbon as the x-coordinate, and the corresponding peak area as the y-coordinate to draw a total carbon standard curve.

A.5.1 Method summary

In the nitric acid medium, the chloride in the sample and the added silver nitrate generate silver chloride precipitate. Through the comparison with the standard turbidity solution, determine the chloride content in the sample.

A.5.2 Reagents and materials

A.5.2.1 Nitric acid solution: 1 + 2.

A.5.2.2 Silver nitrate solution: 17 g/L.

A.5.2.3 Chloride standard solution: 1 mL of solution contains 0.010 mg of chlorine (Cl). Use a pipette to transfer-take 10.00 mL of the chloride standard solution prepared in accordance with GB/T 602; place it in a 100 mL volumetric flask. Use water to dilute to the scale, then, shake it well. This solution shall be prepared right before use.

A.5.3 Analytical procedures

Weigh-take 5.00 g ± 0.01 g of sample; place it in a 25 mL colorimetric tube. Add water to a volume of about 20 mL. Then, successively add 2 mL of nitric acid solution and 1 mL of silver nitrate solution. Use water to dilute to the scale, then, shake it well. Place it for 10 min, then, compare it with the standard turbidity solution. The turbidity of the sample solution shall not be greater than the standard turbidity solution.

The preparation of standard turbidity solution: use a pipette to transfer-take 15.00 mL of chloride standard solution; place it in a 25 mL of colorimetric tube. Process it in the same mode and at the same time as the sample.

A.6 Determination of Nitrate (calculated as NO_3)

A.6.1 Reagents and materials

A.6.1.1 Sulfuric acid.

A.6.1.2 Sodium chloride.

A.6.1.3 Indigo solution: weigh-take 0.18 g of indigo sodium disulfonate ($C_{16}H_8O_8N_2S_2Na_2$); dissolve it in 100 mL of water. The period of use is 60 d.

A.6.2 Analytical procedures

Weigh-take 3.48 g ± 0.01 g of sample; place it in a 25 mL colorimetric tube. Add an appropriate amount of water to dissolve and dilute to 10 mL; add 0.005 g of sodium chloride; shake to dissolve it. Add 0.1 mL of indigo solution and 10 mL of sulfuric acid. Within 5 min, if the blue color does not disappear, then, it means that the nitrate (calculated as NO_3) is less than or equal to 0.0005%.

A.7.5 Result calculation

The mass fraction w_2 of sulfate shall be calculated in accordance with Formula (A.3):

$$w_2 = \frac{(\rho_1 - \rho_0) \times 0.1}{m_3 \times 1\,000} \times 100\% \qquad \cdots\cdots (A.3)$$

Where,

ρ_1---the mass concentration of sulfate in the sample solution obtained through the standard curve, expressed in (mg/L);

ρ_0---the mass concentration of sulfate in the blank sample solution obtained through the standard curve, expressed in (mg/L);

0.1---the volume of the sample solution, expressed in (L);

m_3---the mass of the sample, expressed in (g);

1,000---conversion factor.

The mass fraction w_3 of iron (Fe) [or lead (Pb), cadmium (Cd)], expressed in (mg/kg), shall be calculated in accordance with Formula (A.4):

$$w_3 = \frac{(\rho_3 - \rho_2) \times 0.1 \times 1\,000}{m_3} \qquad \cdots\cdots (A.4)$$

Where,

ρ_3---the mass concentration of test element in the sample solution obtained through the standard curve, expressed in (mg/L);

ρ_2---the mass concentration of the test element in the blank sample solution obtained through the standard curve, expressed in (mg/L);

0.1---the volume of the sample solution, expressed in (L);

1,000---conversion factor;

m_3---the mass of the sample, expressed in (g).

The test result 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 what is specified in Table A.4.

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