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National Food Safety Standard - Food Contact Materials and Articles - Determination of Stibium Migration

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National Food Safety Standard - Food Contact Materials and Articles - Determination of Stibium Migration

1 Scope

This Standard specifies the graphite furnace atomic absorption spectrometry, atomic fluorescence spectrometry, inductively coupled plasma mass spectrometry, inductively coupled plasma emission spectrometry, and malachite green spectrophotometry for the determination of stibium migration after food contact materials and articles are soaked in food simulants.

This Standard is applicable to the determination of stibium migration in food contact materials and articles.

Method One -- Graphite furnace atomic absorption spectrometry

2 Principle

Use the food simulant to immerse the parts of food contact materials and articles intended to come into contact with food. Soaking solution is atomized by graphite furnace. The absorption value measured at 231.2 nm is proportional to the cerium content over a range of concentrations. Compare and quantify with the standard series.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method are all superior-grade pure, and the water is Grade two water specified in GB/T 6682.

3.1 Reagents

3.1.1 Ammonium dihydrogen phosphate ($NH_4H_2PO_4$).

3.1.2 Hydrochloric acid (HCl).

3.1.3 Sulfuric acid (H_2SO_4).

3.1.4 Nitric acid (HNO_3).

4 Instruments and equipment

NOTE: All glassware needs to be soaked overnight with nitric acid solution (1+5). Rinse clean with water.

4.1 Atomic absorption spectrometer: with graphite furnace atomizer, stibium hollow cathode lamp.

4.2 Analytical balance: resolution of 0.1 mg.

5 Analysis steps

5.1 Sample pretreatment

According to the expected use of the testing sample and the conditions of use, with the migration test methods and test conditions specified in GB 5009.156 and GB 31604.1, the migration test shall be carried out. After the soaking solution is fully mixed, take some soaking test solution for analysis. If the soaking test solution is neutral or alkaline, add appropriate amount of nitric acid to make the concentration of nitric acid in the test solution about 5% (volume fraction). Perform the sample blank test at the same time.

5.2 Determination

5.2.1 Instrument test conditions

See Table A.1 for instrument test conditions.

5.2.2 Making of standard curve

According to the order of concentration from low to high, pipette 10 μL of standard solution series and 5 μL of ammonium dihydrogen phosphate solution (20 g/L) (the best injection volume can be determined according to the instrument used) into the graphite furnace at the same time. After atomization, determine the absorbance value. Take the standard series concentration as the abscissa, the corresponding absorbance value as the ordinate to draw a standard curve.

NOTE: Based on the sensitivity of the instrument, the linear range, and the actual content of deuterium in the sample solution, the concentration and range of stibium in the standard series solution can be determined.

5.2.3 Sample determination

Under the same experimental conditions as the determination of standard solution, pipette 10 μL of the sample solution and 5 μL of ammonium dihydrogen phosphate solution (20 g/L) (the best injection volume can be determined

10.1.2 Potassium borohydride (KBH_4).

10.1.3 Thiourea (CH_4N_2S).

10.1.4 Ascorbic acid ($C_6H_8O_6$).

10.1.5 Hydrochloric acid (HCl): superior-grade pure.

10.1.6 Reagents for preparing food simulants: according to GB 31604.1.

10.2 Reagent preparation

10.2.1 Hydrochloric acid solution (1+9): measure 50 mL of hydrochloric acid, add 450 mL of water, mix well.

10.2.2 Hydrochloric acid solution (5%): measure 10 mL of hydrochloric acid, add water to 200 mL, and mix well. Prepare before use.

10.2.3 Potassium borohydride alkaline solution (20 g/L): weigh 2.5 g of sodium hydroxide dissolved in water, completely dissolve, add 10 g of potassium borohydride, add water to 500 mL, and mix well. Prepare before use.

10.2.4 Thiourea-ascorbic acid solution: respectively pipette 10 g of thiourea and 10 g of ascorbic acid, dissolve in water and add water and set volume to 100 mL. Prepare before use.

10.2.5 Food simulant: prepare in accordance with the provisions of GB 5009.156.

10.3 Standard product

Metal stibium (Sb, CAS No: 7440-36-0): purity >99.99% or the standard solution certified by the state and granted with a standard substance certificate.

10.4 Preparation of standard solution

10.4.1 Stibium standard stock solution (500 mg/L): see 3.4.1.

10.4.2 Stibium standard intermediate solution (10.0 mg/L): transfer 2.00 mL of stibium standard stock solution (500 mg/L); place in a 100mL volumetric flask; dilute to the scale with hydrochloric acid solution (1+9); mix well.

10.4.3 Stibium standard use solution (0.100 mg/L): accurately transfer 1.00 mL of standard intermediate solution (10.0 mg/L) into a 100 mL volumetric flask; dilute to the scale with water; mix well.

100mL of water, mix well.

17.3 Standard product

Metal strontium (Sb, CAS number: 7440-36-0): purity > 99.99%, or a standard solution that has been certified by the state and granted with a standard substance certificate.

17.4 Preparation of standard solution

17.4.1 Stibium standard stock solution (500 mg/L): same as 3.4.1.

17.4.2 Stibium standard use solution (10.0 mg/L): take 2mL of stibium standard stock solution, dilute to 100mL with sulfuric acid solution (1+5).

18 Instruments and equipment

NOTE: All glassware needs to be soaked overnight with nitric acid solution (1+5). Rinse clean with water for use.

18.1 Spectrophotometer: with 1cm cuvette.

18.2 Analytical balance: resolution of 0.1mg.

19 Analysis steps

19.1 Sample pretreatment

Same with 5.1.

19.2 Determination

19.2.1 Making of standard curve

Take 0 mL, 0.300 mL, 0.400 mL, 0.600 mL, 0.800 mL, 1.00 mL (equivalent to 0 μg, 3.00 μg, 4.00 μg, 6.00 μg, 8.00 μg, 10.0 μg of stibium) of stibium standard use solutions. Respectively place them in separatory funnels added with 4 mL of water and 4 mL of hydrochloric acid solution (5+1). Add 2 drops of stannous chloride solution (12%). Mix well. Place 5min. Add 1mL of sodium nitrite solution (20%). Mix well. And blow the air with a rubber suction ball to chase the brown nitrogen oxide gas in the separatory funnel. Then add 2.5mL of urea solution (1000 g/L). Shake well and mix thoroughly. Put into the solution and no bubbles escape. Add 1mL of malachite green solution (0.2%). Add 10mL of sodium citrate solution (20%). Then add 5mL of isoamyl acetate. Fully shake for 30s. Place for stratification. Discard the aqueous phase. The organic phase passes through a small funnel pre-filled with a little anhydrous sodium sulfate. The

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