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National Food Safety Standard - Food Contact Materials and Products - Determination of Lead and Its Migration

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Foreword

This Standard replaces the determination of lead of its migration of GB/T 5009.62-2003, *Method for analysis of hygienic standard of ceramics for food containers*, GB/T 5009.63-2003, *Method for analysis of hygienic standard of enamel for food containers*, GB/T 5009.72, *Method for analysis of hygienic standard of aluminum-wares for food use*, GB/T 5009.78-2003, *Method for analysis of hygienic standard of papers for food packaging*, GB/T 5009.81-2003, *Method for analysis of hygienic standard of stainless steel food containers and table wares*, GB/T 3534-2002, *Standard testing methods for lead and cadmium release from domestic ceramics*, GB 8058-2003, *Standard permissible limits and testing method for release of lead or cadmium from ceramic cookware*, GB/T 21170-2007, *Glass hollowware – Test method for lead and cadmium release*, SN/T 2597-2010, *Determination of lead, cadmium, chromium, arsenic, antimony, germanium migration quantity in polymer for food contact materials – Inductively coupled plasma atomic emission spectrometry method*, SN/T 2829-2011, *Food contact materials for export – Metal materials – Determination of migrant heavy metals in food simulant – Inductively coupled plasma atomic emission spectrometric method*, SN/T 2886, *Food contact materials for export – Determination of lead and cadmium release in glass container simulants*, and SN/T 2594-2010, *Food contact materials – Determination of lead, cadmium, chromium, and arsenic in cork stoppers by inductively coupled plasma mass spectrometry*.

Compared with GB/T 5009.62-2003, the main changes of this Standard are as follows:

-- it changes the name of the standard into "*National Food Safety Standard – Food Contact Materials and Products – Determination of Lead and Its Migration*";

-- it adds the determination of lead;

-- it adds the graphite furnace atomic absorption spectrometric method;

-- it adds the inductively coupled plasma mass spectrometric method;

-- it adds the inductively coupled plasma atomic emission spectrometric method; and

-- it deletes the dithizone colorimetric method.

National Food Safety Standard – Food Contact Materials and Products – Determination of Lead and Its Migration

1 Application Scope

This Standard specifies the graphite furnace atomic absorption spectrometric method, inductively coupled plasma mass spectrometric method, inductively coupled plasma atomic emission spectrometric method and flame atomic absorption spectrometric method for the determination of lead in paper products and cork stoppers and lead migration after being soaked in the food simulants.

This Standard applies to the determination of lead in paper products and cork stoppers and lead migration in food contact materials and products.

Part I – Determination of Lead

Method I – Graphite Furnace Atomic Absorption Spectrometric Method

2 Principle

Grind the paper products or cork stoppers; use the dry method for digestion; atomize the digestion solution in a graphite furnace; determine absorbency by comparing with the standard series (the absorption value measured at 283.3 nm within a certain concentration range is in direct proportion to the lead content).

3 Reagents and Materials

Unless specified otherwise, all reagents used for this method are guaranteed reagents and the water is water grade II specified in GB/T 6682.

3.1 Reagents

3.1.1 Nitric acid (HNO_3).

3.1.2 Ammonium dihydrogen phosphate ($NH_4H_2PO_4$).

3.1.3 Palladium nitrate [$Pd(NO_3)_2$].

3.2 Preparation of reagents

3.2.1 Nitric acid solution (5 + 95): measure 50 mL of nitric acid; add to 950 L of water; and mix up.

4.4 Analytical balance: sensitivities 0.1 mg and 1 mg.

5 Analytical Procedure

5.1 Digestion of sample

Take an appropriate amount of sample, grind and mix up. Weigh 1 g to 5 g (accurate to 0.001 g) of sample to place into a crucible; first use soft fire to carbonize to smokeless on an adjustable electric hot plate; transfer to muffle furnace at 500˚C for ashing for 6 h to 8 h and then cool down. If any test piece is not ashed completely, add 1 mL of nitric acid to heat on the adjustable electric hot plate with soft fire; repeat it for multiple times until it is digested completely; place it aside for cooling; use nitric acid (1 + 1) to dissolve the ash; transfer to a 25 mL volumetric flask; use a small amount of water to wash the crucible for multiple times; combine the washings into the volumetric flask and dilute to scale; and mix up for standby. Meanwhile, use it as the reagent blank.

5.2 Determination

5.2.1 Apparatus testing conditions

See Table A.1 for the reference conditions.

5.2.2 Establishment of standard curve

In accordance with the sequence of concentrations from low to high, put 10 μL of lead standard serial solution and 5 μL of palladium nitrate solution-ammonium dihydrogen phosphate solution (the sample size can be decided in accordance with the apparatus used) respectively into the graphite furnace at the same time; measure their absorption values after atomization; and establish the standard curve with the concentrations as the horizontal coordinates and the absorption values as the vertical coordinates.

5.2.3 Determination of sample

Place 10 μL of blank solution or sample solution and 5 μL of palladium nitrate solution-ammonium dihydrogen phosphate solution (the sample size can be decided in accordance with the apparatus used) into a graphite furnace at the same time; measure its absorption value after atomization; and determine the absorbance by comparing with the standard series.

6 Expression of Analytical Results

The lead content in the sample is calculated in accordance with the following equation:

$$X = \frac{(\rho - \rho_0) \times V}{m \times 1\ 000} \qquad \cdots\cdots (1)$$

absorbance by comparing with the standard series (the absorption value measured at 283.3 nm within a certain concentration range is in direct proportion to the lead content).

10 Reagents and Materials

Unless specified otherwise, all reagents used for this method are guaranteed reagents and the water is water grade II specified in GB/T 6682.

10.1 Reagents

10.1.1 Nitric acid (HNO_3).

10.1.2 Ammonium dihydrogen phosphate ($NH_4H_2PO_4$).

10.1.3 Reagents required for the preparation of food simulant: as specified in GB 30604.1.

10.2 Preparation of reagents

10.2.1 Food simulant: prepared as specified in GB 5009.156.

10.2.2 Nitric acid (5 + 95): measure 50 mL of nitric acid; add to 950 mL of water; and mix up.

10.2.3 Nitric acid (1 + 9): measure 50 mL of nitric acid; add to 450 mL of water; and mix up.

10.2.4 Ammonium dihydrogen phosphate (20 g/L): weigh 2.0 g of ammonium dihydrogen phosphate; use water to dissolve; and dilute to 100 mL.

10.3 Standard substance

Lead nitrate [$Pb(NO_3)_2$, CAS no.: 10099-74-8]: purity > 99.99% or the lead standard solution of a certain concentration which has been certified by a national certification institution and awarded the certificate of standard substance.

10.4 Preparation of standard solutions

10.4.1 Lead standard stock solution (1 000 mg/L): weigh accurately 1.598 5 g (accurate to 0.000 1 g); use a small amount of nitric acid solution (1 + 9) to dissolve; transfer to a 1000 mL volumetric flask; add water to scale and mix up.

10.4.2 Lead standard intermediate solution (1.00 mg/L): absorb 1.00 mL of lead standard stock solution to place into a 1 000 mL volumetric flask; add nitric acid solution (5 + 95) to scale; and mix up.

10.4.3 Lead standard serial solution: absorb respectively 0 mL, 0.500 mL, 1.00 mL, 2.00 mL, 4.00 mL and 6.00 mL of lead standard intermediate solution (1.00 mg/L) to

17 Reagents and Materials

Unless specified otherwise, all reagents used for this method are guaranteed reagents and the water is water grade II specified in GB/T 6682.

17.1 Reagents

17.1.1 Nitric acid (HNO_3).

17.1.2 Reagents required for the preparation of food simulant: as specified in GB 30604.1.

17.2 Preparation of reagents

17.2.1 Food simulant: prepared as specified in GB 5009.156.

17.2.2 Nitric acid (5 + 95): measure 50 mL of nitric acid; add to 950 mL of water; and mix up.

17.2.3 Nitric acid (1 + 9): measure 50 mL of nitric acid; add to 450 mL of water; and mix up.

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Lead nitrate [$Pb(NO_3)_2$, CAS no.: 10099-74-8]: purity > 99.99% or the lead standard solution of a certain concentration which has been certified by a national certification institution and awarded the certificate of standard substance.

17.4 Preparation of standard solutions

17.4.1 Lead standard stock solution (1 000 mg/L): weigh accurately 1.598 5 g (accurate to 0.000 1 g); use a small amount of nitric acid solution (1 + 9) to dissolve; transfer to a 1000 mL volumetric flask; add water to scale and mix up.

17.4.2 Lead standard intermediate solution (10.0 mg/L): absorb 1.00 mL of lead standard stock solution to place into a 1 00 mL volumetric flask; add nitric acid solution (5 + 95) to scale; and mix up.

17.4.3 Lead standard serial solution: absorb respectively 0 mL, 0.500 mL, 1.00 mL, 2.00 mL, 3.00 mL and 4.00 mL of lead standard intermediate solution (10.0 mg/L) to place into a 10.0 mL volumetric flask; add corresponding food simulants to scale and mix up. Then the concentrations of the lead standard serial solution are respectively 0 mg/L, 0.500 mg/L, 1.00 mg/L, 2.00 mg/L, 3.00 mg/L and 4.00 mg/L.

NOTE The specific concentrations of lead in the standard solution series can be decided in accordance with the sensibility and linearity range of the instrument and the actual lead content in the sample. If the selected food simulants are neutral or alkaline, it needs to add an appropriate amount of nitric acid to make sure the nitric acid concentration in the solution is approximately 5% (volume fraction).