



GB 5009.12—2017 National Food Safety Standard Determination of Lead in Foods

GB 5009.12—2017 食品安全国家标准
食品中铅的测定

NHFPC & CFDA

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National Standard of the People's Republic of China

GB 5009.12-2017

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食品安全国家标准

食品中铅的测定

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Preface

This standard replaces the method for determination of lead in GB 5009.12-2010 “National Food Safety Standard Determination of Lead in Foods”, GB/T 20380.3-2006 “Starch and Derived Products-Heavy Metals Content-Part 3: Determination of Lead Content by Atomic Absorption Spectrometry with Electrothermal Atomization”, GB/T 23870-2009 “Determination of Pb in Propolis-Microwave Digestion-Graphite Furnace Atomic Absorption Spectrophotometry”, GB/T 18932.12-2002 “Method for the Determination of Potassium, Sodium, Calcium, Magnesium, Zinc, Iron, Copper, Manganese, Chromium, Lead, Cadmium Contents in Honey-Atomic Absorption Spectrometry”, NY/T 1100-2006 “Determination of Lead, Cadmium in Rice-Graphite Furnace Atomic Absorption Spectrometry”, and SN/T 2211-2008 “Determination of Lead, Cadmium in Royal Jelly-Graphite Furnace Atomic Absorption Spectrometry Method”.

Compared with GB 5009.12-2010, the main changes of this standard are as follows:

——in the pretreatment method, the wet digestion and digestion by pressure digestion tank have been retained, the dry ashing and ammonium persulfate ashing method have been deleted and the microwave digestion has been added;

——the graphite furnace atomic absorption spectrometry method has been retained as the method I, using ammonium dihydrogen phosphate-palladium nitrate solution as the matrix modifier; the flame atomic absorption spectrometry has been retained as the method III; and the dithizone colorimetry has been retained as the method IV;

——the inductively coupled plasma mass spectrometry has been added as the method II;

——the hydride generation atomic fluorescence spectrometry and single sweep polarography have been deleted;

——the Annexes for temperature programming for microwave digestion, and instrument reference conditions for graphite furnace atomic absorption spectrometry and flame atomic absorption spectrometry have been added.

National Food Safety Standard

Determination of Lead in Foods

1 Scope

This standard specifies the methods for determination of lead in foods: graphite furnace atomic absorption spectrometry, inductively coupled plasma mass spectrometry, flame atomic absorption spectrometry and dithizone colorimetry.

This standard applies to the lead determination in all kinds of foods.

Method I Graphite Furnace Atomic Absorption Spectrometry

2 Principles

After digestion, the test sample is atomized by graphite furnace and the absorbance is determined at 283.3 nm. In certain concentration range, the absorbance value of lead is proportional to lead content, and the quantitative analysis can be conducted by comparing with standard series.

3 Reagents and Materials

Unless otherwise specified, the reagents used in this method all refer to guaranteed reagents and the water shall be the Grade 2 water specified in GB/T 6682.

3.1 Reagents

3.1.1 Nitric acid (HNO_3).

3.1.2 Perchloric acid (HClO_4).

3.1.3 Ammonium dihydrogen phosphate ($\text{NH}_4\text{H}_2\text{PO}_4$).

3.1.4 Palladium nitrate [$\text{Pd}(\text{NO}_3)_2$].

3.2 Preparation of reagents

3.2.1 Nitric acid solution (5+95): measure 50 mL of nitric acid, slowly add it to 950 mL of water, and mix it well.

3.2.2 Nitric acid solution (1+9): measure 50 mL of nitric acid, slowly add it to 450 mL of water, and mix it well.

3.2.3 Ammonium dihydrogen phosphate-palladium nitrate solution: weigh 0.02 g of palladium nitrate, add a small amount of nitric acid solution (1+9) to dissolve, and then add 2 g of ammonium dihydrogen phosphate and after dissolution, dilute it to 100 mL with nitric acid solution (5+95) and mix it well.

3.3 Standard substances

Lead nitrate [$\text{Pb}(\text{NO}_3)_2$, CAS No.: 10099-74-8]: with the purity of greater than 99.99%. Or the lead standard solution at a certain concentration which is nationally certified with Reference Material Certificate.

3.4 Preparation of standard solutions

3.4.1 Lead standard stock solution (1000 mg/L): accurately weigh 1.5985 g (accurate to 0.000 1g) of lead nitrate, dissolve it with a small amount of nitric acid solution (1+9), and then transfer it to a 1000-mL volumetric flask, add water to the volume and mix it well.

3.4.2 Lead standard intermediate solution (1.00 mg/L): accurately pipette 1.00 mL of lead standard stock solution (1000 mg/L) into a 1000-mL volumetric flask, add nitric acid solution (5+95) to the volume, and mix it well.

3.4.3 Lead standard series solutions: respectively pipette 0 mL, 0.500 mL, 1.00 mL, 2.00 mL, 3.00 mL and 4.00 mL of lead standard intermediate solution (1.00 mg/L) into 100-mL volumetric flasks, add nitric acid solution (5+95) to the volume, and mix it well. The mass concentration of this lead standard series solution is 0 $\mu\text{g/L}$, 5.00 $\mu\text{g/L}$, 10.0 $\mu\text{g/L}$, 20.0 $\mu\text{g/L}$, 30.0 $\mu\text{g/L}$ and 40.0 $\mu\text{g/L}$, respectively.

Note: The mass concentration of lead in standard series solutions can be determined according to the sensitivity of the instrument and the actual content of lead in the sample.

4 Apparatus and Equipment

Note: All glassware and polytetrafluoroethylene digestion inner tanks are required to be soaked overnight with nitric acid solution (1+5), repeatedly rinsed with tap water, and finally washed clean with water.

4.1 Atomic absorption spectrometer: equipped with graphite furnace atomizer and lead hollow cathode lamp.

4.2 Analytical balance: with the sensitivity of 0.1 mg and 1 mg.

4.3 Adjustable electric furnace.

4.4 Adjustable electric heating plate.

4.5 Microwave digestion system: equipped with polytetrafluoroethylene digestion inner tank.

4.6 Constant temperature drying oven.

4.7 Pressure digestion tank: equipped with polytetrafluoroethylene digestion inner tank.

5 Analysis Procedures

5.1 Preparation of test sample

Note: During sampling and preparation, the test sample shall be prohibited from contamination.

5.1.1 Grains and bean samples

After the removal of impurities, grains and beans are smashed and stored in the plastic bottle for use.

5.1.2 Vegetables, fruits, fish, meat and other samples

Such samples are washed clean with water, and dried in the air; edible parts are taken and processed into homogenate, and stored in plastic bottles for use.

5.1.3 Beverage, wine, vinegar, soy sauce, edible vegetable oil, liquid milk and other liquid samples

Shake such samples well.

5.2 Pretreatment of test sample

5.2.1 Wet digestion

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