National Standard of the People's Republic of China

GB 5009.12-2017

National Standards For Food Safety
Determination Of Lead In Food

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1 Scope

This standard specifies the graphite furnace atomic absorption spectrometry, inductively coupled plasma mass spectrometry, flame atomic absorption spectrometry, and dithizone colorimetric method for the determination of lead content in food.

This standard is applicable to the determination of lead content in various types of food.

The First Method: Graphite Furnace Atomic Absorption Spectrometry

2 Principles

After the sample is digested and atomized in a graphite furnace, the absorbance is measured at 283.3nm. The absorbance value of lead is directly proportional to the lead content within a certain concentration range, and is quantitatively compared to the standard series.

3 Reagents and Materials

Unless otherwise specified, the reagents used in this method are all high-grade pure, and the water is Grade II water as specified in GB/T6682.

3.1 Reagents

3.1.1 Nitric acid (HNO_3).

3.1.2 Perchlorate ($HClO_4$).

3.1.3 Ammonium dihydrogen phosphate ($NH_4H_2PO_4$).

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

3.2 Reagent preparation

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

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

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

3.3 Standards

Lead nitrate [Pb (NO3) 2, CAS No. 10099-74-8]: Purity>99.99%. Or a certain concentration of lead standard solution certified by the state and awarded with a standard substance certificate.

3.4 Preparation of standard solution

3.4.1 Lead standard stock solution (1000mg/L): Accurately weigh 1.5985g (accurate to 0.0001g) of lead nitrate, dissolve with a small amount of nitric acid solution (1+9), transfer to a 1000mL volumetric flask, add water to the mark, and mix well.

3.4.2 Lead standard intermediate solution (1.00mg/L): Accurately absorb 1.00mL of lead standard reserve solution (1000mg/L) into a 1000mL volumetric flask, add nitric acid solution (5+95) to the mark, and mix well.

3.4.3 Lead standard series solution: Take 0mL, 0.500mL, 1.00mL, 2.00mL, 3.00mL, and 4.00mL of lead standard intermediate solution (1.00mg/L) into a 100mL volumetric flask, add nitric acid solution (5+95) to the mark, and mix well. The mass concentrations of this lead standard series solution are 0 μg/L, 5.00 μg/L, 10.0 μg/L, 20.0 μg/L, 30.0 μg/L and 40.0 μg/L.

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

4 Instruments and equipment

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

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

4.2 Analytical balance: sensitivity 0.1mg and 1mg.

4.3 Adjustable electric furnace.

4.4 Adjustable electric heating plate.

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

4.6 Constant temperature drying oven.

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

5 Analysis steps

5.1 Sample preparation

Note: During the sampling and sample preparation process, sample contamination should be avoided.

5.1.1 Grain and legume samples

After removing impurities, the sample is crushed and stored in a plastic bottle.

5.1.2 Samples of vegetables, fruits, fish, meat, etc

Wash the sample with water, air dry it, take the edible part, make a homogenate, and store it in a plastic bottle

5.1.3 Liquid samples of beverages, wine, vinegar, soy sauce, edible vegetable oil, liquid milk, etc

Shake the sample well.

5.2 Sample Preparation

5.2.1 Wet digestion

Weigh 0.2g~3g of solid sample (accurate to 0.001g) or accurately transfer

0.500mL~5.00mL of liquid sample into a graduated digestion tube, add 10mL of nitric acid and 0.5mL of perchloric acid, and digest in an adjustable electric furnace (reference conditions: 120 ℃/0.5h~1h; rise to 180 ℃/2h~4h, rise to 200 ℃~220 ℃).

If the digestive solution is brownish brown, add a small amount of nitric acid and digest until white smoke appears. The digestive solution is colorless, transparent, or slightly yellow. Take out the digestive tube, cool it, and bring it to a constant volume of 10mL with water. Mix well for later use. Simultaneously perform reagent blank tests. Alternatively, a conical flask can be used for wet digestion on an adjustable electric heating plate according to the above operation method.

5.2.2 Microwave digestion

Weigh 0.2g~0.8g of solid sample (accurate to 0.001g) or accurately transfer 0.500mL~3.00mL of liquid sample into a microwave digestion tank, add 5mL of nitric acid, and digest the sample according to the microwave digestion steps. The digestion conditions refer to Appendix A.After cooling, remove the digestion tank and drive the acid to about 1mL on an electric heating plate at 140 ℃~160 ℃. After the digestion tank is cooled, transfer the digestion solution to a 10mL volumetric flask, wash the digestion tank 2-3 times with a small amount of water, combine the washing solution into the volumetric flask and bring it to volume with water, mix well and set aside. Simultaneously perform reagent blank tests.

5.2.3 Pressure Tank Digestion

Weigh 0.2g~1g of solid sample (accurate to 0.001g) or accurately transfer 0.500mL~5.00mL of liquid sample into the digestion tank, and add 5mL of nitric acid.Cover the inner cover, tighten the stainless steel jacket, place it in a constant temperature drying oven, and maintain it for 4-5 hours at 140 ℃~160 ℃.

After cooling, slowly loosen the outer tank, remove the digestion inner tank, and place it on an adjustable electric heating plate to drive the acid to about 1mL at 140 ℃~160 ℃.After cooling, transfer the digestive solution to a 10mL volumetric flask, wash the inner tank and lid 2-3 times with a small amount of water, merge the washing solution into the volumetric flask, and bring it to volume with water. Mix

well for later use. Simultaneously perform reagent blank tests.

5.3 Determination

5.3.1 Instrument reference conditions

Adjust to the optimal state based on the performance of each instrument. Refer to Appendix B for reference conditions.

5.3.2 Production of standard curves

According to the order of mass concentration from low to high, the 10μL lead standard series solution and 5μL ammonium dihydrogen phosphate - palladium nitrate solution (the best sample amount can be determined according to the instrument used) were injected into the graphite furnace at the same time, and their absorbance values were measured after atomization. The mass concentration was taken as the horizontal coordinate and the absorbance value was taken as the vertical coordinate to make a standard curve.

5.3.3 Determination of sample solution

Under the same experimental conditions as the standard solution, apply 10 μ L blank solution or sample solution and 5 μ L ammonium dihydrogen phosphate palladium nitrate solution (the optimal injection amount can be determined based on the instrument used) is simultaneously injected into a graphite furnace, and its absorbance value is measured after atomization, and compared quantitatively with the standard series.

6 Expression of analysis results

Calculate the lead content in the sample according to formula(1):

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

In the formula

X - The content of lead in the sample, in milligrams per kilogram or milligrams per liter (mg/kg or mg/L);

ρ - The mass concentration of lead in the sample solution, in micrograms per liter（ μg/L);

ρ 0- Mass concentration of lead in blank solution, in micrograms per liter（ μg/L);

V - The constant volume of the sample digestion solution, in milliliters (mL);

M - Sample weighing or volume taken, in grams or milliliters (g or mL);

1000- Conversion coefficient

When the lead content is ≥ 1.00 mg/kg (or mg/L), the calculation results shall retain three significant digits; When the lead content is less than 1.00 mg/kg (or mg/L), retain two significant digits in the calculation results.

7 Precision

The absolute difference between two independent measurement results obtained under repeatability conditions shall not exceed 20% of the arithmetic mean

8 Others

When the sample size is 0.5g (or 0.5mL) and the constant volume is 10mL, the detection limit of the method is 0.02mg/kg (or 0.02mg/L), and the quantitative limit is 0.04mg/kg (or 0.04mg/L).

Appendix A

Microwave digestion heating program

Steps	Setting temperature(℃)	Heating up time(Min)	Holding time(Min)
1	120	5	5
2	160	5	10
3	180	5	10

Appendix B

Reference conditions for graphite furnace atomic absorption spectrometry instruments

The reference conditions for graphite furnace atomic absorption spectrometry instruments are shown in Table B.1.

Table B.1 Reference conditions for graphite furnace atomic absorption spectrometry instruments

Elements	Wavelength (nm)	Slit (nm)	Lamp current	Drying	Ashing	Atomization
Lead(Pb)	283.3	0.5	8~12	85 ℃~120 ℃/40s~50s	750 ℃/20s~30s	2300 ℃/4s~5s