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National food safety standard - Determination of lead in foods

食品安全国家标准　食品中铅的测定

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Table of Contents

National food safety standard - Determination of lead in foods

1 Scope

This Standard specifies the methods for the determination of lead in foods by graphite furnace atomic absorption spectrometry, inductively coupled plasma mass spectrometry and flame atomic absorption spectrometry.

This Standard applies to the determination of lead in foods.

Method I – Graphite furnace atomic absorption spectrometry

2 Principle

After digestion treatment, the sample is atomized in a graphite furnace and the absorbance is measured at 283.3 nm. Within a certain concentration range, the absorbance value of lead is proportional to the lead content and can be compared quantitatively with the standard series.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method are guaranteed reagents, and the water is grade-II water as 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 ($NH_4H_2PO_4$).

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

3.1.5 Ammonium acetate (CH_3COONH_4).

3.1.6 Sodium acetate (CH_3COONa).

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; mix well.

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

3.2.3 Nitric acid solution (1+99): Measure 10 mL of nitric acid; slowly add it to 990 mL of water; mix well.

3.2.4 Sodium acetate solution (2 mol/L): Weigh 164.0 g of sodium acetate; add water to dissolve; adjust the volume to 1 000 mL.

3.2.5 Ammonium acetate solution (1 mol/L): Weigh 77.1 g of ammonium acetate; add water to dissolve; adjust the volume to 1 000 mL.

3.2.6 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 it; then, add 2 g of ammonium dihydrogen phosphate; after dissolution, use nitric acid solution (5+95) to adjust the volume to 100 mL; mix well.

3.3 Standard

Lead nitrate [$Pb(NO_3)_2$, CAS number: 10099-74-8]: purity >99.99%, or lead standard solution certified by the state and granted with a reference material certificate.

3.4 Preparation of standard solution

3.4.1 Lead standard stock solution (1 000 mg/L): Accurately weigh 1.598 5 g (accurate to 0.000 1 g) of lead nitrate; use a small amount of nitric acid solution (1+9) to dissolve it; transfer it to a 1 000 mL volumetric flask; add water to the mark; mix well.

3.4.2 Lead standard intermediate solution (10.0 mg/L): Accurately draw 1.00 mL of lead standard stock solution (1 000 mg/L) into a 100 mL volumetric flask; use nitric acid solution (5+95) to adjust the volume to the mark; mix well.

3.4.3 Lead standard working solution (1.00 mg/L): Accurately draw 10.00 mL of lead standard intermediate solution (10.0 mg/L) into a 100 mL volumetric flask; use nitric acid solution (5+95) to adjust the volume to the mark; mix well.

3.4.4 Lead standard series solution: Respectively draw 0 mL, 0.2 mL, 0.5 mL, 1.0 mL, 2.0 mL and 4.0 mL of lead standard working solution (1.00 mg/L) into 100 mL volumetric flasks; add nitric acid solution (5+95) to the mark; mix well. The mass concentrations of this lead standard series of solutions are 0 μg/L, 2.0 μg/L, 5.0 μg/L, 10.0 μg/L, 20.0 μg/L and 40.0 μg/L, respectively.

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

5.1.2 Liquid samples

For samples of soft drinks, alcoholic beverages, condiments, etc., shake well.

5.1.3 Semi-solid samples

Mix well.

5.2 Sample pretreatment

5.2.1 Wet digestion

Weigh 0.2 g ~ 3 g (accurate to 0.001 g) of solid sample or accurately transfer 0.50 mL ~ 5.00 mL of liquid sample into a graduated digestion tube. For samples containing ethanol or carbon dioxide, first heat at low temperature on an electric hot plate to remove ethanol or carbon dioxide; add 10 mL of nitric acid and 0.5 mL of perchloric acid; put a few glass beads; digest it on an adjustable electric furnace (reference conditions: 120 °C/0.5 h ~ 1 h; heated to 180 °C/2 h ~ 4 h, heated to 200 °C ~ 220 °C). If the digestion solution is brown, add a small amount of nitric acid and digest until white smoke is emitted and the digestion solution is colorless, transparent or slightly yellow. Remove the remaining acid until almost dry; stop digestion; cool and add water to adjust the volume to 10 mL or 25 mL; mix well and set aside. Do a reagent blank test at the same time. Alternatively, use an Erlenmeyer flask and perform wet digestion on an adjustable electric hot plate according to the above operation methods.

Note: The added volume of nitric acid and perchloric acid can be adjusted according to the actual situation.

5.2.2 Microwave digestion

Weigh 0.2 g ~ 2 g (accurate to 0.001 g) of the solid sample or accurately transfer 0.50 mL ~ 3.00 mL of the liquid sample into the microwave digestion tank. For samples containing ethanol or carbon dioxide, first heat at low temperature on an electric hot plate to remove the ethanol or carbon dioxide; add 5 mL ~ 10 mL of nitric acid (the amount of nitric acid used can be adjusted according to the weighing amount and properties of the sample); digest the sample according to the operating steps of microwave digestion. For digestion conditions, refer to Appendix A. After cooling, take out the digestion tank and remove the remaining acid on the electric hot plate at 140 °C ~ 160 °C until it is almost dry. After the digestion tank cools off, transfer the digestion solution to a 10 mL or 25 mL volumetric flask; use a small amount of water to wash the digestion tank 2 ~ 3 times; combine the cleaning mixture in the volumetric flask and use water to adjust the volume to the mark; mix well and set aside. Do a reagent blank test at the same time.

5.2.3 Pressure tank digestion

Weigh 0.2 g ~ 2 g (accurate to 0.001 g) of the solid sample or accurately transfer 0.50 mL ~ 5.00 mL of the liquid sample into the digestion inner tank. For samples containing ethanol or carbon dioxide, first heat at low temperature on an electric hot plate to remove the ethanol or carbon dioxide; add 5 mL ~ 10 mL of nitric acid (the amount of nitric acid used can be adjusted according to the weighing amount and properties of the sample). Close the inner cover; screw the stainless-steel outer cover tightly; put it in a constant-temperature drying oven; keep it at 140 °C ~ 160 °C for 4 h ~ 5 h. After cooling, slowly unscrew the outer tank, take out the digestion inner tank, and place it on an adjustable electric hot plate to remove the remaining acid at 140 °C ~ 160 °C until it is almost dry. After cooling, transfer the digestion solution to a 10 mL or 25 mL volumetric flask; use a small amount of water to wash the inner tank and inner cover 2 ~ 3 times; combine the cleaning mixture in the volumetric flask; use water to dilute to the mark; mix well and set aside. Do a reagent blank test at the same time.

Note: High-salt foods such as table salt, soy sauce, pickled foods, hot pot soup bases and instant noodle salt packets can be desalinated. For details, see Appendix B.

5.3 Determination

5.3.1 Apparatus reference conditions

See Appendix C for apparatus reference conditions.

5.3.2 Preparation of standard curve

In order of mass concentration from low to high, add 10 μL of lead standard series solution and 5 μL of ammonium dihydrogen phosphate-palladium nitrate solution (the optimal injection volume and optimal matrix modifier can be determined according to the instrument used, and samples passing through the solid-phase extraction column do not need to be added with matrix modifier) into the graphite furnace at the same time, and measure their absorbance values after atomization. Make a standard curve with the mass concentration as the abscissa and the absorbance value as the ordinate.

5.3.3 Determination of sample solution

Under the same experiment conditions as the measurement of the standard solution, inject 10 μL of blank solution or sample solution and 5 μL of ammonium dihydrogen phosphate-palladium nitrate solution (the optimal injection volume can be determined according to the instrument used) into the graphite furnace at the same time, which can be omitted for the sample passing through the solid-phase extraction column; measure its absorbance value after atomization; compare it quantitatively with the standard series.

6 Expression of analysis results

The content of lead in the sample is calculated according to Formula (1).

10.2.2 Nitric acid solution (1+9): Measure 50 mL of nitric acid; slowly add it to 450 mL of water; mix well.

10.2.3 Ammonium sulfate solution (300 g/L): Weigh 30 g of ammonium sulfate; use water to dissolve and dilute to 100 mL; mix well.

10.2.4 Ammonium citrate solution (250 g/L): Weigh 25 g of ammonium citrate; use water to dissolve and dilute to 100 mL; mix well.

10.2.5 Bromothymol blue aqueous solution (1 g/L): Weigh 0.1 g of bromothymol blue; use water to dissolve and dilute to 100 mL; mix well.

10.2.6 DDTC solution (50 g/L): Weigh 5 g of DDTC; use water to dissolve and dilute to 100 mL; mix well.

10.2.7 Ammonia solution (1+1): Take 100 mL of ammonia; add 100 mL of water; mix well.

10.2.8 Hydrochloric acid solution (1+11): Take 10 mL of hydrochloric acid; add 110 mL of water; mix well.

10.3 Standard

Lead nitrate [$Pb(NO_3)_2$, CAS number: 10099-74-8]: purity >99.99%, or lead standard solution certified by the state and granted with a reference material certificate.

10.4 Preparation of standard solution

10.4.1 Lead standard stock solution (1 000 mg/L): Accurately weigh 1.598 5 g (accurate to 0.000 1 g) of lead nitrate; use a small amount of nitric acid solution (1+9) to dissolve it; transfer it to a 1 000 mL volumetric flask; add water to the mark; mix well.

10.4.2 Lead standard working solution (10.0 mg/L): Accurately draw 1.00 mL of lead standard stock solution (1 000 mg/L) into a 100 mL volumetric flask; add nitric acid solution (5+95) to the mark; mix well.

11 Instruments and apparatuses

Note: All glassware needs to be soaked in nitric acid solution (1+5) or nitric acid solution (1+4) overnight, rinsed repeatedly with tap water, and finally rinsed with water and dried.

11.1 Atomic absorption spectrometer: equipped with flame atomizer and accompanied by lead hollow cathode lamp.

11.2 Analytical balance: The sensitivity is 0.1 mg and 1 mg, respectively.

11.3 Adjustable electric furnace and adjustable electric hot plate.

Appendix B

Desalted sample operation steps

B.1 Sample digestion

B.1.1 Wet digestion

Follow the steps of "Weigh the solid sample...the digestion solution is colorless, transparent or slightly yellow; remove the remaining acid until almost dry" in 5.2.1; after cooling, use sodium acetate solution (2 mol/L) to wash the digestion tank 2 ~ 3 times; combine the cleaning mixture in a 25 mL volumetric flask; use sodium acetate solution (2 mol/L) to adjust the volume to the mark; mix well and set aside (the pH of the solution after dilution is 4.5 ~ 6.5). Do a reagent blank test at the same time.

B.1.2 Microwave digestion

Follow the steps of "Weigh the solid sample...remove the remaining acid on the electric hot plate at 140 °C ~ 160 °C until it is almost dry" in 5.2.2; after cooling, use sodium acetate solution (2 mol/L) to wash the digestion tank 2 ~ 3 times; combine the cleaning mixture in a 25 mL volumetric flask; use sodium acetate solution (2 mol/L) to adjust the volume to the mark; mix well and set aside (the pH of the solution after dilution is 4.5 ~ 6.5). Do a reagent blank test at the same time.

B.1.3 Pressure tank digestion

Follow the steps of "Weigh the solid sample... place it on an adjustable electric hot plate to remove the remaining acid at 140 °C ~ 160 °C until it is almost dry" in 5.2.3; after cooling, use sodium acetate solution (2 mol/L) to wash the inner tank and inner cover 2 ~ 3 times; combine the cleaning mixture in a 25 mL volumetric flask; use sodium acetate solution to adjust the volume to the mark; mix well and set aside (the pH of the solution after dilution is 4.5 ~ 6.5). Do a reagent blank test at the same time.

B.2 Separation of lead

B.2.1 Activation of solid-phase extraction column

Pipette 10 mL of nitric acid solution (1+99) through the column at a flow rate of 5 mL/min; then, use 5 mL of water and 5 mL of ammonium acetate solution (1 mol/L) to pass through the column at a flow rate of 5 mL/min.

B.2.2 Adsorption and desorption of lead

Take 25 mL of the reagent blank solution and the above sample solution respectively; pass them through the column at a flow rate of 5 mL/min; then, use 5 mL of ammonium acetate solution (1 mol/L) to wash the column; then, use 10 mL of water to wash away

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