NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 5009.12-2023

National food safety standard - Determination of lead in foods

Issued on: September 6, 2023 **Implemented on: September 6, 2024**

Issued by: NHC; SAMR

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Foreword

This Standard replaces GB 5009.12-2017 Food safety national standard - Determination of lead in food.

Compared with GB 5009.12-2017, the major changes of this Standard are as follows.

-- Add the pretreatment method for samples that need to be desalted in Method 1 - Graphite furnace atomic absorption spectrometry;

-- Remove Method 4 - Dithizone colorimetry;

-- Modify the detection limit and quantitation limit of Method 1 - Graphite furnace atomic absorption spectrometry.

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 1 - 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.

3.2.1 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(NO3)2, CAS number. 10099-74-8]. purity >99.99%, or lead standard solution certified by the state and granted with a reference material certificate.

4 Instruments and apparatuses

Note. All glassware and polytetrafluoroethylene digestion inner tanks need 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.

4.1 Atomic absorption spectrometer. equipped with graphite furnace atomizer and

accompanied by lead hollow cathode lamp.

5.1.2 Liquid samples

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

5.1.3 Semi-solid samples

Mix well.

5.3.1 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.

6 Expression of analysis results

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

7 Precision

When the lead content in the sample is greater than 1 mg/kg, the absolute difference between the two independent measurement results obtained under repeatability conditions shall not exceed 10% of the arithmetic mean; when it is less than or equal to

8 Others

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

For samples such as raw milk, pasteurized milk, sterilized milk, fruit and vegetable juices and their beverages [excluding fruit and vegetable juices containing berries and small fruits, as well as their beverages, and concentrated fruit and vegetable juices (pulp)], liquid infant and young child formula foods, etc., when the sample volume is 2 g (or 2 mL) and the constant volume is 10 mL, the detection limit of the method is 0.005 mg/kg (or 0.005 mg/L), and the quantitation limit is 0.01 mg/kg (or 0.01 mg/L).

9 Principle

After the sample is processed, lead ions form a complex with sodium diethyldithiocarbamate (DDTC) under certain pH conditions, which is extracted and separated by 4-methyl-2-pentanone (MIBK) and introduced into the atomic absorption spectrometer. After flame atomization, measure the absorbance at 283.3 nm. Within a certain concentration range, the absorbance value of lead is proportional to the lead