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NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 5009.16-2023

National food safety standard - Determination of tin in foods

食品安全国家标准　食品中锡的测定

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State Administration for Market Regulation.

Table of Contents

National food safety standard - Determination of tin in foods

1 Scope

This Standard specifies the methods for the determination of tin in foods by hydride atomic fluorescence spectrometry, inductively coupled plasma mass spectrometry and inductively coupled plasma-atomic emission spectrometry.

This Standard applies to the determination of tin in foods.

Method I – Hydride atomic fluorescence spectrometry

2 Principle

After the sample is digested, tin hydride (SnH_4) is generated under the action of sodium borohydride (or potassium borohydride), and is brought into the atomizer by the carrier gas for atomization. Under the irradiation of a tin hollow cathode lamp, the ground state tin atoms are excited to an upper state. When deactivated and returned to the ground state, they emit fluorescence of a characteristic wavelength. The fluorescence intensity is proportional to the tin content and can be compared quantitatively with a standard series of solutions.

3 Reagents and materials

Unless otherwise specified, all the reagents used in this method are analytical reagents, and the water used is grade-II water as specified by GB/T 6682.

3.1 Reagents

3.1.1 Sulfuric acid (H_2SO_4): guaranteed reagent.

3.1.2 Nitric acid (HNO_3): guaranteed reagent.

3.1.3 Perchloric acid ($HClO_4$): guaranteed reagent.

3.1.4 Thiourea (CH_4N_2S).

3.1.5 Ascorbic acid ($C_6H_8O_6$).

3.1.6 Sodium borohydride ($NaBH_4$) or potassium borohydride (KBH_4).

3.1.7 Sodium hydroxide (NaOH) or potassium hydroxide (KOH).

3.2 Preparation of reagents

3.2.1 Nitric acid-perchloric acid mixed solution (4+1): Measure 400 mL of nitric acid and 100 mL of perchloric acid, and mix well.

3.2.2 Sulfuric acid solution (1+9): Measure 100 mL of sulfuric acid, slowly add 900 mL of water, and mix well.

3.2.3 Thiourea + ascorbic acid solution: Weigh 15.0 g of thiourea and 15.0 g of ascorbic acid, use water to dissolve and dilute to 100 mL, and mix well. Prepare when necessary.

3.2.4 Sodium hydroxide solution (5 g/L): Weigh 5.0 g of sodium hydroxide, use water to dissolve and dilute to 1 000 mL, and mix well.

3.2.5 Sodium borohydride solution (7 g/L): Weigh 7.0 g of sodium borohydride, use sodium hydroxide solution (5 g/L) to dissolve and dilute to 1 000 mL, and mix well. Prepare when necessary.

Note: Alternatively, use potassium borohydride as the reducing agent (dissolved and prepared with potassium hydroxide solution) in this method, and adjust the concentration of sodium borohydride or potassium borohydride in the reducing agent according to the sensitivity of the instrument (7 g/L ~ 20 g/L).

3.3 Standard

Metal tin (Sn) standard product: purity ≥99.99%. Or tin standard solution of a certain concentration that is certified by the state and awarded a reference material certificate.

3.4 Preparation of standard solution

3.4.1 Tin standard solution (1.00 mg/mL): Accurately weigh 0.100 0 g of metal tin standard; place it in a small beaker; add 10.0 mL of sulfuric acid; cover with a watch glass; heat until the tin is completely dissolved; remove the watch glass; continue to heat until thick white smoke appears; cool; slowly add 50 mL of water; transfer to a 100 mL volumetric flask; use sulfuric acid solution (1+9) to wash the beaker several times; merge the cleaning mixture into the volumetric flask; dilute to the mark; mix well .

3.4.2 Tin standard intermediate solution (10.0 mg/L): Accurately draw 1.00 mL of tin standard solution (1.00 mg/mL) into a 100 mL volumetric flask; use sulfuric acid solution (1+9) to dilute to the mark; mix well. Store at 0 °C ~ 5 °C, valid for 4 weeks.

3.4.3 Tin standard working solution (1.00 mg/L): Accurately draw 10.0 mL of tin standard intermediate solution (10.0 mg/L) into a 100 mL volumetric flask; use sulfuric acid solution (1+9) to dilute to the mark; mix well. Store at 0 °C ~ 5 °C, valid for 4 weeks.

3.4.4 Tin standard series solutions: Respectively take 0 mL, 0.500 mL, 2.00 mL, 3.00 mL, 4.00 mL, and 5.00 mL of tin standard working solution (1.00 mg/L) into 25 mL volumetric flasks; respectively add 5.00 mL, 4.50 mL, 3.00 mL, 2.00 mL, 1.00 mL, and 0.00 mL of sulfuric acid solution (1+9); add 2.0 mL of thiourea + ascorbic acid solution;

Take the entire edible content (including the edible liquid part) and make it into a homogenate or uniform powder.

5.1.2 Liquid samples

For samples such as soft drinks and liquid condiments, shake well.

5.1.3 Semi-solid sample

Mix well.

5.2 Sample digestion

5.2.1 Weigh 1 g ~ 5 g (accurate to 0.001 g) of the sample or accurately transfer 1.00 mL ~ 5.00 mL of the liquid sample into a glass digestion vessel. Heat samples containing ethanol or carbon dioxide at 90 °C for 30 minutes to remove the ethanol or carbon dioxide. Add 10.0 mL of nitric acid-perchloric acid mixed solution (4+1); add 1.0 mL of sulfuric acid; digest on an electric hot plate or graphite digestion device. If the digestion solution turns brown and black during the digestion process, take it out immediately and let it cool. Add nitric acid or nitric acid-perchloric acid mixed solution (4+1) appropriately before continuing the digestion. When the digestion solution emits white smoke and appears colorless, transparent or slightly yellow, continue to heat until the remaining volume is about 1 mL; remove and cool; use water to dilute to 50 mL; mix well and set aside to obtain a sample solution. According to the tin content in the sample solution, use water to dilute the sample solution to an appropriate concentration when necessary, and add a certain amount of sulfuric acid solution (1+9) during the dilution process, so that the sulfuric acid concentration of the final sample solution is the same as that of the standard series solution. Do a blank test at the same time.

5.2.2 Accurately pipette 10.0 mL of blank solution and sample solution respectively; place them in a 25 mL volumetric flask; add 3.0 mL of sulfuric acid solution (1+9); add 2.0 mL of thiourea + ascorbic acid solution; then, use water to adjust the volume to 25 mL; mix well to obtain blank determination solution and sample determination solution.

5.3 Apparatus reference conditions

Negative high voltage 380 V; lamp current 70 mA; atomization temperature 850 °C; shielding gas flow 1 200 mL/min; carrier gas flow 500 mL/min; measurement method the standard curve method; reading method the peak area; delay time 1 s; reading time 15 s; liquid-adding time 8 seconds; injection volume 2.0 mL.

5.4 Preparation of the standard curve

After the instrument is preheated and stabilized, inject the standard series solution into the atomic fluorescence spectrometer; measure the fluorescence intensity of tin; draw a standard curve with the mass concentration of tin in the standard series solution as the abscissa and the fluorescence intensity as the ordinate.

8 Others

When the sampling volume is 1 g (or 1 mL), the constant volume of the sample solution is 50 mL, the detection limit of the method is 0.8 mg/kg (or 0.8 mg/L), and the quantitation limit of the method is 2.5 mg/kg (or 2.5 mg /L).

Method II – Inductively coupled plasma mass spectrometry

9 Principle

The sample is digested by nitric acid + hydrochloric acid, and the tin in it is dissolved and maintained stable under the action of hydrochloric acid. It is measured by an inductively coupled plasma mass spectrometer and qualitatively determined by the specific mass number (mass-to-charge ratio, m/z) of the tin element. The ratio of mass spectrum signal response value of tin element to internal standard element is proportional to the concentration of tin element for quantitative analysis.

10 Reagents and materials

Unless otherwise stated, the reagents used in this method are guaranteed reagents, and the water is Grade-I water as specified in GB/T 6682.

10.1 Reagents

10.1.1 Nitric acid (HNO_3): guaranteed reagent or of higher purity.

10.1.2 Hydrochloric acid (HCl): guaranteed reagent or of higher purity.

10.1.3 Argon (Ar): Argon (≥99.995%) or liquid argon.

10.1.4 Helium (He): Helium (≥99.995%).

10.2 Preparation of reagents

10.2.1 Hydrochloric acid solution (5+95): Measure 50 mL of hydrochloric acid; slowly add 950 mL of water; mix well.

10.2.2 Nitric acid-hydrochloric acid mixed solution (5+1+94): Measure 50 mL of nitric acid; slowly add 940 mL of water; mix well; then, slowly add 10 mL of hydrochloric acid; mix well.

10.3 Standard

10.3.1 Tin standard solution (1 000 mg/L): Use the tin reference solution certified by the state and awarded a reference material certificate.

10.3.2 Internal standard element stock solution (1 000 mg/L): Use any single element or multi-element internal standard stock solution such as rhodium (Rh) and rhenium (Re) that has been certified by the state and awarded a reference material certificate.

10.4 Preparation of standard solution

10.4.1 Tin standard intermediate solution (10.0 mg/L): Accurately draw 1.00 mL of tin standard solution (1 000 mg/L) into a 100 mL volumetric flask; use hydrochloric acid solution (5+95) to dilute to the mark; mix well. Store at 0 °C ~ 5 °C, valid for 4 weeks.

10.4.2 Tin standard working solution (1.00 mg/L): Accurately draw 10.0 mL of tin standard intermediate solution (10.0 mg/L) into a 100 mL volumetric flask; use hydrochloric acid solution (5+95) to dilute to the mark; mix well. Store at 0 °C ~ 5 °C, valid for 4 weeks.

10.4.3 Tin standard series solutions: Respectively take 0 mL, 0.250 mL, 0.500 mL, 1.00 mL, 2.00 mL and 5.00 mL of tin standard working solution (1.00 mg/L) into 100 mL volumetric flasks; use nitric acid-hydrochloric acid mixed solution (5 + 1 + 94) to adjust the volume to the mark; mix well. The mass concentrations of this tin standard series of solutions are 0 μg/L, 2.50 μg/L, 5.00 μg/L, 10.0 μg/L, 20.0 μg/L, and 50.0 μg/L, respectively. Prepare when necessary.

Note: The mass concentration range of tin element in the standard series solution can be determined based on the sensitivity of the instrument and the actual content of tin in the sample.

10.4.4 Internal standard working solution: Take an appropriate amount of internal standard element stock solution; use nitric acid-hydrochloric acid mixed solution (5+1+94) to dilute it step by step to prepare an internal standard working solution of appropriate concentration. The internal standard solution can be added manually to the standard series and sample solutions quantitatively, or it can be added online by the instrument. After the internal standard is mixed with the sample solution, the reference mass concentration of the internal standard is approximately 10 μg/L ~ 100 μg/L.

11 Instruments and apparatuses

11.1 Inductively coupled plasma mass spectrometer.

11.2 Analytical balance: the sensitivity is 1mg.

11.3 Adjustable temperature-controlled electric furnace or electric hot plate: the temperature control range is 80 °C ~ 100 °C.

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

11.5 Sample crushing equipment: homogenizer, high-speed crusher.

12 Analysis steps

12.1 Sample preparation

Same as 5.1.

12.2 Sample digestion

Weigh 0.2 g ~ 0.5 g (accurate to 0.001 g; the sampling amount can be appropriately increased to 1 g for samples containing more moisture) of the sample, or accurately transfer 0.500 mL ~ 3.00 mL of the liquid sample into the microwave digestion inner tank. For samples containing ethanol or carbon dioxide, heat at 90 °C for 30 minutes to remove ethanol or carbon dioxide. Add 5 mL ~ 7 mL of nitric acid; cover and leave overnight at room temperature; add 1 mL of hydrochloric acid and tighten the cover as soon as possible; digest according to the standard operating procedures of the microwave digestion instrument (see Appendix A, Table A.1 for digestion reference conditions). After cooling, take it out, and slowly open the cover to exhaust; use a small amount of water to rinse the inner lid; place the digestion tank on an adjustable temperature-controlled electric furnace or electric hot plate; heat at 90 °C for 30 minutes; use water to adjust the volume to 50 mL; mix well and set aside. Do a blank test at the same time.

12.3 Apparatus reference conditions

Tin isotope ^{118}Sn, internal standard element ^{103}Rh or ^{185}Re; radio frequency power 1 550 W; plasma gas flow 15.0 L/min; carrier gas flow 1.00 L/min; auxiliary gas flow 0.90 L/min; sampling depth 10 mm; analysis mode the normal mode or collision/reaction cell mode; repeated for 2 ~ 3 times; flushing time 60 seconds.

Note: Some tin compounds are easily hydrolyzed to form insoluble matter remaining in the pipeline. In order to avoid hydrolysis and reduce flushing time, nitric acid-hydrochloric acid mixed solution (5+1+94) or hydrochloric acid solution (volume fraction 1% ~ 5%) should be used to flush the pipeline.

12.4 Preparation of standard curve

Inject the tin standard series solutions into the inductively coupled plasma mass spectrometer respectively; measure the signal response values of the tin element and the internal standard element. Draw a standard curve with the mass concentration of tin element as the abscissa and the ratio of the signal response value of tin element to internal standard element as the ordinate.

12.5 Test of sample solution

Inject the blank solution and sample solution into the inductively coupled plasma mass spectrometer respectively; measure the signal response values of tin element and

15 Others

When the sampling volume is 0.5 g (or 0.5 mL) and the fixed volume is 50 mL, the detection limit of the method is 0.08 mg/kg (or 0.08 mg/L), and the quantitation limit is 0.25 mg/kg (or 0.25 mg/L).

Method III – Inductively coupled plasma-atomic emission spectrometry

16 Principle

The sample is digested by nitric acid + hydrochloric acid, and the tin in it is dissolved and maintained stable under the action of hydrochloric acid. It is measured by an inductively coupled plasma emission spectrometer. The wavelength of the characteristic spectral line of the tin element is used for qualitative identification; the tin element spectral line intensity response value being proportional to the element concentration is used for quantitative analysis.

17 Reagents and materials

Unless otherwise stated, the reagents used in this method are guaranteed reagents, and the water is Grade-I water as specified in GB/T 6682.

17.1 Reagents

17.1.1 Nitric acid (HNO_3): guaranteed reagent or of higher purity.

17.1.2 Hydrochloric acid (HCl): guaranteed reagent or of higher purity.

17.1.3 Argon (Ar): Argon (≥99.995%) or liquid argon.

17.2 Preparation of reagents

17.2.1 Hydrochloric acid solution (5+95): Measure 50 mL of hydrochloric acid; slowly add 950 mL of water; mix well.

17.2.2 Nitric acid-hydrochloric acid mixed solution (5+1+94): Measure 50 mL of nitric acid; slowly add 940 mL of water; mix well; then, slowly add 10 mL of hydrochloric acid; mix well.

17.3 Standard

Tin standard solution (1 000 mg/L): Use the tin reference solution certified by the state and awarded a reference material certificate.

17.4 Preparation of standard solution

Note: Some tin compounds are easily hydrolyzed to form insoluble matter remaining in the pipeline. In order to avoid hydrolysis and reduce flushing time, nitric acid-hydrochloric acid mixed solution (5+1+94) or hydrochloric acid solution (volume fraction 1% ~ 5%) should be used to flush the pipeline.

19.4 Preparation of standard curve

Inject the tin standard series solution into the inductively coupled plasma emission spectrometer; measure the intensity response value of the tin element analysis spectrum line; draw a standard curve with the mass concentration of the tin element as the abscissa and the analysis spectrum line intensity response value as the ordinate.

19.5 Determination of sample solution

Inject the blank solution and sample solution into the inductively coupled plasma emission spectrometer respectively; measure the intensity response value of the tin element analysis spectral line; obtain the mass concentration of tin element in the solution according to the standard curve. If the tin content in the sample solution exceeds the range of the standard curve, use nitric acid-hydrochloric acid mixed solution (5+1+94) to dilute it.

20 Expression of analysis results

The content of tin in the sample is calculated according to Formula (3).

$$X = \frac{(\rho - \rho_0) \times V \times f}{m} \quad \cdots\cdots (3)$$

Where:

X – the content of tin in the sample, in milligrams per kilogram (mg/kg) or milligrams per liter (mg/L);

ρ – the mass concentration of tin in the sample solution, in milligrams per liter (mg/L);

ρ_0 – the mass concentration of tin in the blank solution, in milligrams per liter (mg/L);

V – the constant volume of the sample solution, in milliliters (mL);

f – dilution factor of the sample solution;

m – sample weighing amount or pipetting volume, in grams (g) or milliliters (mL);

Three significant figures shall be kept for the calculation result.

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