NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 5009.16-2023

National food safety standard - Determination of tin 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 5009.16-2014 National Food Safety Standard -

Determination of Tin in food.

Compared with GB 5009.16-2014, the major changes of this Standard are as follows.

-- Add inductively coupled plasma mass spectrometry as method II;

-- Add inductively coupled plasma-atomic emission spectrometry as method III;

-- Remove phenofluorenone colorimetry;

-- Modify the scope of application of method I - hydride atomic fluorescence spectrometry, by adding the method detection limit.

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.

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.

3.2.1 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.

3.3 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.

3.4.1 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;

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.

4.2 Adjustable temperature control electric hot plate. temperature control range 90 °C

~ 250 °C.

5.1.2 Liquid samples

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

5.1.3 Semi-solid sample

Mix well.

5.2.1 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.

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;

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.

5.5 Determination of sample solution

Respectively inject the blank determination solution and the sample determination solution into the atomic fluorescence spectrometer;

6 Expression of analysis results

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

7 Precision

The absolute difference of 2 independent test results obtained under repeatability cannot exceed 10% of the arithmetic mean value.

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).

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.