

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC **OF CHINA**

中华人民共和国国家标准

GB 5009.11-2014

National Standard for Food Safety Determination of total arsenic and inorganic arsenic in the food

食品安全国家标准 食品中总砷及无机砷的测定

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Foreword

This standard shall replace *Determination of Total Arsenic and Inorganic Arsenic in the Food* GB/T 6009.11-2003.

This standard has the following major changes compared to GB/T 6009.11-2003:

The standard name is revised into National Standard for Food Safety-Determination of Total Arsenic and Inorganic Arsenic in the Food.

Canceled Gutzeit method for determination of total arsenic and reduction assay for hydroboron in the food, and atomic fluorescence spectrometry and silver diethyl-dithiocarbamate method for determination of inorganic arsenic in the food.

Added inductively coupled plasma mass spectrometer (ICP-MS) for determination of total arsenic;

Added liquid chromatogram-atomic fluorescence spectroscopy(LC-AFS) and inductively coupled plasma mass spectromete (LC-ICP-MS) for determination of inorganic arsenic.

National Standard for Food Safety

Determination of total arsenic and inorganic arsenic in the food

1. Scope

Chapter I of this standard specifies determination method for total arsenic in the food; and Chapter II of this standard specifies liquid chromatogram-atomic fluorescence spectroscopy and inductively coupled plasma mass spectrometer (LC-ICP-MS) for determination of inorganic arsenic.

Method I, II and II in Chapter I of this standard is applicable for determination of total arsenic in various food, and Chapter II applied to determination of inorganic arsenic in rice, aquatic livestock, frumentum accessory food for infant, canned accessory food for infant, including arsenate and arsenite.

Chapter I Determination of total arsenic

Method I Inductively coupled plasma mass spectrometer

2. Principle

Samples are processed as the sample solution by acid digestion, sample solution is atomized to deliver to ICP torch by the carrier, and transferred into charged ions after evaporation, dissociation, atomization and ionization etc., and then enter the mass spectrometer through the ion acquisition system, and the mass spectrometer make separation according to the mass-to-charge ratio. For certain mass-to-charge ratio, the signal intensity of the mass spectrum is direct proportional to the number of ions entering the mass spectrometer, which means the sample concentration is direct proportional to the signal intensity of the mass spectrum. It shall determine arsenic in the sample solution through measurement of the signal intensity of the mass spectrum.

3. Reagent and materials

Note: except as otherwise stated, all reagents used in this method is guarantee reagent, and water is Grade I specified in GB/T 6682.

3.1 Reagent

- 3.1.1 Nitric acid(HNO₃): grade MOS (special high purity chemicals for electronic industry), Grade BV(III).
- 3.1.2 Hydrogen peroxide (H₂O₂).
- 3.1.3 Mass spectrum tuned liquid: Li, Y, Ce, Ti, Co, recommended use concentration to be 10ng/mL.
- 3.1.4 Internal standard stock solution: Ge, concentration to be 100 ug/mL.
- 3.1.5 Sodium hydroxide (NaOH).

3.2 Reagent preparation

- 3.2.1 Salpeter solution (2+98): weigh 30 mL of nitric acid, slowly poure into 980 mL of water, and mix.
- 3.2.2 Internal standard solution GE or Y (1.0 ug/mL): weigh 1.0 mL of internal standard solution, dilute it with salpeter solution (2+98), and meter volume to 100mL.
- 3.2.3 Sodium hydroxide solution (100g/L): weigh 10.0 g of sodium hydroxide, dilute it with water and meter volume to 100 mL.

3.3 Standard product

Arsenic trioxide (As₂O₃) standard product: purity \geq 99.5%.

3.4 Preparation of standard solution

- 3.4.1 Arsenic standard stock solution (100 mg/L, calculated according to As): accurately weigh 0.0132 g of arsenic trioxide dried at 100 centigrade degree for 2 hours, add 1mL of sodium hydroxide solution (100g/L) and a little water to dissolve, transfer it into 100 mL of volumetric flask, add a proper amount of hydrochloric acid to adjust its acidity into neutral, and dilute with water to the scale. Store at 4 centigrade degree away from light, with one year of storage period. Or purchase standard solution substance approved by the state and authorized with standard substances certificate.
- 3.4.2 Arsenic standard use solution (1.00 mg/L, calculated according to As): accurately weigh 1.00 mL of arsenic standard stock solution (1.00 mg/L), place into 100 mL of volumetric flask, dilute it with salpeter solution (2+98), and meter volume to the scale. Prepare for immediate use.

4. Instrument and equipment



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