



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

GB 5009.93-2017

**National food safety standard - Determination of selenium in
foods**

(English Translation)

Issued on: April 6, 2017

Implemented on: October 6, 2017

Issued by: National Health and Family Planning Commission of the

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Foreword

This Standard replaces GB 5009.93-2010 "National food safety standard -Determination of selenium in foods", GB/T 21729-2008 "Tea - Determination of selenium content", SN/T 0860-2000 "Method for the determination of selenium in canned mushroom for export - Fluorometry" and SN/T 0926-2000 "Method for the determination of selenium in tea for import and export - Fluorimetry".

Compare this Standard with GB 5009.93-2010, the main changes are as follows.

- RETAIN the hydride atomic fluorescence spectrometry as Method I, fluorescence spectrophotometry as Method II;
- ADD the inductively coupled plasma mass spectrometry as Method III.

National food safety standard -Determination of selenium in foods

1 Scope

This Standard specifies the determination of selenium content in foods by hydride atomic fluorescence spectrometry, fluorescence spectrometry and inductively coupled plasma mass spectrometry.

This Standard applies to the determination of selenium in all types of foods.

Method I -- Hydride atomic fluorescence spectrometry

2 Principle

After the sample is acid-heated and digested, in the 6 mol/L hydrochloric acid medium, the hexavalent selenium in the sample is reduced to tetravalent selenium. Sodium borohydride or potassium borohydride is used as the reducing agent to reduce the tetravalent selenium in the hydrochloric acid medium to hydrogen selenide, which is introduced by carrier gas (argon) into the atomizer for atomization.

3 Reagents and materials

Unless otherwise indicated, the reagents used in this method are analytical reagents, and the water is the Grade 2 water as specified in GB/T 6682.

3.1 Reagents

3.1.1

Nitric acid (HNO₃). excellent reagent.

3.1.2

Perchloric acid (HClO₄). excellent reagent.

3.1.6

Sodium borohydride (NaBH₄). excellent reagent.

3.1.7

Potassium ferricyanide [K₃Fe(CN)₆].

3.2.1 Nitric acid - perchloric acid mixed acid (9 + 1). MIX 900 mL of nitric acid with 100

mL of perchloric acid.

3.2.2 Sodium hydroxide solution (5 g/L). WEIGH 5 g of sodium hydroxide; DISSOLVE

in 1000 mL of water; MIX well.

3.3 Standard

Selenium standard solution. 1000 mg/L, or selenium standard solution at a certain concentration certified by the state and awarded the standard substance certificate.

3.4.1 Selenium standard intermediate solution (100 mg/L). accurately PIPETTE 1.00

mL of selenium standard solution (1000 mg/L) in a 10 mL volumetric flask; ADD hydrochloric acid solution (5 + 95) to the mark; MIX well.

4 Instruments and equipment

NOTE: All glassware and polytetrafluoroethylene digestion inner tank must be soaked in nitric acid solution (1 + 5) overnight, washed repeatedly with tap water, and finally rinsed with water.

4.1

Atomic fluorescence spectrometer. with selenium hollow cathode lamp.

4.4

Microwave digestion system. with polytetrafluoroethylene digestion inner tank.

5.1 Sample preparation

NOTE: Sample contamination shall be avoided during sampling and preparation processes.

5.1.1 Cereal, bean samples

The sample, after the debris are removed and being crushed, is stored in plastic bottles.

5.2.1 Wet digestion

WEIGH 0.5 g ~ 3 g (to the nearest 0.001 g) of solid sample or accurately PIPETTE 1.00 mL ~ 5.00 mL of liquid sample; PLACE in a conical flask; ADD 10 mL of nitric acid - perchloric acid mixed acid (9 + 1) and a few glass beads; COVER the surface dish for cold digestion overnight. The next day, HEAT on the electric hot plate, and timely ADD nitric acid. When the solution becomes clear and colorless and accompanied by white smoke, continue to HEAT until the remaining volume is about 2 mL.

5.2.2 Microwave digestion

WEIGH 0.2 g ~ 0.8 g (to the nearest 0.001 g) of solid sample or accurately PIPETTE 1.00 mL ~ 3.00 mL of liquid sample; PLACE in the digestive tube; ADD 10 mL of nitric acid and 2 mL of hydrogen peroxide; SHAKE to mix well. DIGEST in the microwave digestion instrument, and the recommended microwave digestion conditions are shown in Annex A (set digestion conditions according to different instruments).

5.3.1 Instrument reference conditions

Adjust the instruments to the best condition according to their performance. Reference conditions. negative high voltage 340 V; lamp current 100 mA; atomization temperature 800 °C; furnace height 8 mm; carrier gas flow rate 500 mL/min; shielding gas flow rate 1000 mL/min; standard curve method of measurement method; peak area of reading method; delay time 1 s; reading time 15 s; solution adding time 8 s;

sample injection volume 2 mL.

6 Expression of analysis results

The content of selenium in the sample is calculated according to equation (1).

7 Precision

The absolute difference between two independent determination results obtained under repeatability conditions shall not exceed 20 % of the arithmetic mean.

8 Others

When the sample volume is 1 g (or 1 mL) and the constant volume is 10 mL, the limit of detection is 0.002 mg/kg (or 0.002 mg/L) and the limit of quantification is 0.006 mg/kg (or 0.006 mg/L) for this method.

9 Principle

The sample is digested with mixed acid to convert the selenium compound to inorganic selenium Se^{4+} .

10 Reagents and materials

Unless otherwise specified, the reagents used in this method are analytical reagents, and the water is Grade 2 water as specified in GB/T 6682.