GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 5009.268-2025

National Food Safety Standard – Determination of Multi-Elements in Foods

食品安全国家标准 食品中多元素的测定

Issued on: March 16, 2025 **Implemented on: September 16, 2025**

Issued by: National Health Commission of the People's Republic of China;

State Administration for Market Regulation.

Table of Contents

National Food Safety Standard – Determination of Multi-Elements in Foods

1 Scope

The Part I of this Standard specifies the inductively coupled plasma mass spectrometry and inductively coupled plasma emission spectrometry methods for the determination of multiple elements in foods; and the Part II specifies the inductively coupled plasma emission spectrometry method for the determination of multiple elements in nutritional fortifiers of compound foods.

The Method I of the Part I is applicable to the determination of lithium, boron, sodium, magnesium, aluminum, phosphorus, sulfur, potassium, calcium, titanium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, arsenic, selenium, rubidium, strontium, molybdenum, cadmium, tin, antimony, barium, mercury, thallium and lead in foods; and the Method II of the Part I is applicable to the determination of aluminum, arsenic, boron, barium, calcium, cadmium, cobalt, chromium, copper, iron, potassium, magnesium, manganese, molybdenum, sodium, nickel, phosphorus, lead, sulfur, selenium, tin, strontium, titanium, vanadium and zinc in foods.

The Part II is applicable to the determination of calcium, copper, iron, potassium, magnesium, manganese, sodium, phosphorus and zinc in nutritional fortifiers of compound foods with minerals as raw materials, with or without auxiliary materials.

Part I Determination of Multi-Elements in Foods

Method I Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

2 Principle

After digestion, the specimen is measured by inductively coupled plasma mass spectrometry. The qualitative analysis is based on the specific mass number (mass-to-charge ratio, *m*/*z*) of the element; and the quantitative analysis is based on the intensity ratio of the mass spectrum signal of the element to be measured and the mass spectrum signal of the internal standard element, which is proportional to the concentration of the element to be measured.

3 Reagents and Materials

Unless otherwise specified, all reagents used in this Method are of guaranteed reagents; and water is Grade-1 water specified in GB/T 6682.

3.1 Reagents

3.1.1 Nitric acid (HNO_3).

3.1.2 Nitric acid with high purity (HNO_3): The content of the target element in the reagent is less than 1 μg/L. For trace detection, nitric acid with high purity should be used.

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

3.1.4 Helium (He): Helium (≥99.995%).

3.1.5 L-cysteine hydrochloride monohydrate ($C_3H_{10}ClNO_3S$, CAS No.: 7048-04-6): ≥98%, chromatographically pure.

3.1.6 Gold (Au) element solution: The mass concentration is 1,000 mg/L.

3.1.7 Isopropanol (C_3H_8O, CAS No.: 67-63-0): Chromatographically pure.

3.1.8 Hydrochloric acid (HCl).

3.2 Preparation of reagents

3.2.1 Nitric acid solution (5+95)

Take 50 mL of nitric acid, slowly add it into 950 mL of water and mix well.

3.2.2 Mercury standard dilution solution

3.2.2.1 Mercury standard dilution solution A: Take 2 g of L-cysteine hydrochloride monohydrate and dissolve it to 1,000 mL with nitric acid solution (5+95).

3.2.2.2 Mercury standard dilution solution B: Take 0.2 mL of gold (Au) element solution and dilute it to 1,000 mL with nitric acid solution (5+95).

3.3 Standard solution

3.3.1 Single element standard stock solution (1,000 mg/L or 100 mg/L): lithium, boron, sodium, magnesium, aluminum, phosphorus, sulfur, potassium, calcium, titanium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, arsenic, selenium, rubidium, strontium, molybdenum, cadmium, tin, antimony, barium, thallium, lead. Use single element standard stock solution certified by the state and awarded with standard substance/standard sample certificate. Multi-element mixed standard stock solution can also be used.

3.3.2 Mercury standard stock solution (1,000 mg/L or 100 mg/L): Use single element standard stock solution certified by the state and awarded with standard substance/standard sample certificate. Standard series needs to be prepared separately.

3.3.3 Single element standard internal standard stock solution (1,000 mg/L): Single element standard stock solutions such as scandium, germanium, rhodium, indium, rhenium, bismuth, etc.; and multi-element mixed standard internal standard stock solutions can also be used.

NOTE: When using single element standards to prepare mixed standard solutions and internal standard solutions, it is necessary to pay attention to the impurity content of the target element in the single element standard.

3.4 Preparation of standard solutions

3.4.1 Mixed standard solution: Take an appropriate amount of single element standard stock solution or multi-element mixed standard stock solution; dilute it step by step with nitric acid solution (5+95) to prepare a mixed standard solution series. For the preparation method and mass concentration of each element, see Tables A.1 and A.2.

3.4.2 Mercury standard solution: Take an appropriate amount of mercury standard stock solution; dilute it step by step with mercury standard diluent (A or B) to prepare a standard solution series. The mass concentration is shown in Table A.3.

3.4.3 Internal standard solution: Take an appropriate amount of single-element internal standard stock solution or multi-element mixed standard internal standard stock solution; and use nitric acid solution (5+95) to prepare an internal standard solution of appropriate concentration. The concentration of the internal standard solution is shown in A.2.

NOTE: Isopropanol can be appropriately added to the internal standard solution to reduce the sensitization effect of elements such as arsenic and selenium caused by incomplete sample digestion (see Table A.4 for preparation method).

4 Instruments and Equipment

4.1 Inductively coupled plasma mass spectrometer (ICP-MS).

4.2 Balance: With sensitivity of 0.001 g.

4.3 Microwave digester.

4.4 Pressure digester.

4.5 Constant temperature drying oven.

4.6 Temperature-controlled electric heating plate or adjustable electric furnace.

instrument requirements, it can be supplemented with water); cover it and leave it for 1 h or overnight. Tighten the tank cover (for specimens with high tin content, add 1 mL of hydrochloric acid before covering it); and digest according to the standard operating procedures of the microwave digestion instrument (see Table B.1 for digestion reference conditions). After cooling, take it out; slowly open the can lid to vent; rinse the inner lid with a small amount of water. Place the digestion can on a temperature-controlled hot plate or in an ultrasonic water bath, heat at 100°C for 30 min or ultrasonically degas for 2 min~5 min; make constant volume to 25 mL or 50 mL with water; mix well and set aside. Perform a blank test at the same time.

NOTE: To avoid element loss caused by excessive pressure during digestion, it should use a high-pressure microwave digestion container.

5.2.2 Pressure tank digestion method

Weigh 0.2 g ~ 2.0 g of specimen (accurate to 0.001 g, the specimen with high water content can be appropriately increased to 5.0 g), or accurately pipette 0.50 mL ~ 5.00 mL of specimen into the digestion inner tank. For specimens containing ethanol or carbon dioxide, first heat them on a hot plate at low temperature to remove the ethanol or carbon dioxide. Add 3 mL ~ 5 mL of nitric acid; leave them for 1 h or overnight; tighten the stainless-steel jacket (for specimens with high tin content, add 1 mL of hydrochloric acid before covering it); place them in a constant temperature drying oven for digestion (see Table B.1 for digestion reference conditions); digest at 160 °C ~170 °C for 4 h. After cooling, slowly loosen the stainless-steel jacket; take out the digestion inner tank; heat them on a temperature-controlled hot plate or in an ultrasonic water bath at 100 °C for 30 min or ultrasonically degas for 2 min ~5 min. Use water to make constant volume to 25 mL or 50 mL; mix well and set aside. Perform a blank test at the same time.

5.3 Instrument reference conditions

5.3.1 Instrument operating conditions: See Table B.2 for instrument operating conditions; See Table B.3 for element analysis modes.

NOTE: For instruments without a suitable interference elimination mode, interference correction equations are required to correct the measurement results. See Table B.4 for interference correction equations for vanadium, arsenic, selenium, molybdenum, cadmium, lead and other elements.

5.3.2 Determination reference conditions: After tuning the instrument to meet the measurement requirements, edit the measurement method and select the corresponding internal standard element according to the properties of the element to be measured. See Table B.5 for the *m*/*z* of the element to be measured and the internal standard element.

5.4 Drawing of standard curve

Introduce the mixed standard solution and mercury standard solution into the inductively coupled plasma mass spectrometer; determine the signal response values of the element to be measured and the internal standard element; and draw a standard curve with the concentration and mix well.

10.2.2 Nitric acid + perchloric acid (10+1): Take 10 mL of perchloric acid; slowly add it to 100 mL of nitric acid; and mix well.

10.3 Standard solution

10.3.1 Single element standard stock solution (1,000 mg/L or 10,000 mg/L): aluminum, arsenic, boron, barium, calcium, cadmium, cobalt, chromium, copper, iron, potassium, magnesium, manganese, molybdenum, sodium, nickel, phosphorus, lead, sulfur, selenium, tin, strontium, titanium, vanadium, zinc. Use single element standard stock solution certified by the state and awarded with standard substance/standard sample certificate, or multi-element mixed standard stock solution.

10.3.2 Preparation of standard solutions: Accurately pipette an appropriate amount of single-element standard stock solution or multi-element mixed standard stock solution; and dilute it step by step with nitric acid solution (5 +95) to prepare a series of mixed standard solutions. The mass concentration of each element is shown in Table A.5.

11 Instruments and Equipment

11.1 Inductively coupled plasma optical emission spectrometer (ICP-OES).

11.2 Balance: With sensitivity of 0.01 g or 0.001 g.

11.3 Microwave digestion instrument.

11.4 Pressure digester.

11.5 Constant temperature drying oven.

11.6 Temperature-controlled hot plate or adjustable electric furnace.

11.7 Muffle furnace.

11.8 Specimen crushing equipment: homogenizer, high-speed crusher, etc.

11.9 Graphite digestion device.

12 Analysis Steps

12.1 Preparation of specimen

The same as 5.1.

12.2 Digestion of specimen

12.2.1 Microwave digestion method

The same as 5.2.1.

12.2.2 Pressure tank digestion method

The same as 5.2.2.

12.2.3 Wet digestion method

Weigh 0.5 g ~ 5.0 g (accurate to 0.001 g, the specimen with high water content can be appropriately increased to 10.0 g) of specimen, or accurately pipette 2.00 mL~10.0 mL of the specimen into a glass or polytetrafluoroethylene digestion vessel. For specimens containing ethanol or carbon dioxide, first heat them on a hot plate at low temperature to remove the ethanol or carbon dioxide; add 10 mL of nitric acid + perchloric acid (10+1) mixed solution; and digest on a hot plate or graphite digestion device. If the digestion solution turns brown-black during the digestion process, a small amount of mixed acid shall be appropriately added until white smoke is emitted and the digestion solution is colorless and transparent or slightly yellow. Cool it; make constant volume to 25 mL or 50 mL with water; and mix it for later use. Perform a blank test at the same time.

NOTE: For specimens with high tin content, it is recommended to add 1 mL of hydrochloric acid when making the constant volume.

12.2.4 Dry digestion method (except for elements such as arsenic, cadmium, lead, sulfur, selenium and tin)

Weigh 1.0 g ~ 5.0 g (accurate to 0.01 g, the specimen with high water content can be appropriately increased to 15.0 g) of specimen, or accurately pipette 10.0 mL~15.0 mL of the specimen into a crucible; carbonize it on an electric furnace with a low fire until it stops smoking; and then place it in a muffle furnace at 500℃ ~550℃ for ash 5 h~8 h; and cool. If the ash is not thorough and there are black carbon particles, add a little nitric acid to moisten it after cooling; dry it on the electric hot plate; move it into the muffle furnace and continue to ash it into white ash; cool it. Take it out; add 10 mL of nitric acid solution to dissolve it; and make constant volume to 25 mL or 50 mL with water; mix it well for later use. Perform a blank test at the same time.

12.3 Instrument reference conditions

Optimize the instrument operating conditions so that the sensitivity and other indicators of the elements to be measured meet the analysis requirements; edit the determination method; select the appropriate analytical spectrum line for each element to be measured. See B.3.1 for the instrument operation reference conditions; and see Table B.6 for the recommended analytical spectrum lines of the elements to be measured.

12.4 Drawing of standard curve

Part II Determination of Multiple Elements in Nutritional Fortifiers of Compound Foods

16 Principle

After the specimen is dissolved in nitric acid, it is measured by inductively coupled plasma emission spectrometer. The qualitative analysis is carried out based on the element's characteristic emission spectrum line wavelength; and the quantitative analysis is based on the signal intensity of the element's spectrum line proportional to the element concentration.

17 Reagents and Materials

Unless otherwise specified, the reagents used in this method are all guaranteed reagents; and the water is Grade-1 water specified in GB/T 6682.

17.1 Reagents

17.1.1 Nitric acid (HNO_3).

17.1.2 Hydrochloric acid (HCl).

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

17.2 Preparation of reagents

17.2.1 Nitric acid solution (10+90): Take 100 mL of nitric acid; slowly add it to 900 mL of water; and mix well.

17.2.2 Nitric acid solution (4+96): Take 40 mL of nitric acid; slowly add it to 960 mL of water; and mix well.

17.3 Standard solution

17.3.1 Element standard stock solution (1,000 mg/L or 10,000 mg/L): Calcium, copper, iron, potassium, magnesium, manganese, sodium, phosphorus, zinc. Use single element standard stock solution certified by the state and awarded with standard material/standard sample certificate; and multi-element mixed standard stock solution can also be used.

17.3.2 Preparation of standard solution: Accurately pipette an appropriate amount of single element standard stock solution or multi-element mixed standard stock solution; and dilute it step by step with nitric acid solution (4+96) to prepare a series of mixed standard working solutions. The mass concentration of each element is shown in Table C.1 of Appendix C.

18 Instruments and Equipment

18.1 Inductively coupled plasma optical emission spectrometer (ICP-OES).

18.2 Analytical balance: With sensitivity of 0.000 1 g.

18.3 Ultrasonic generator.

18.4 High-speed crusher.

19 Analysis Steps

19.1 Preparation of specimen

For uniform powdered samples of nutritional fortifiers of compound foods, weigh them directly. For non-powdered samples, weigh them after being crushed uniformly by a high-speed crusher.

19.2 Pretreatment of specimen

Weigh 0.5 g ~ 1.0 g of the specimen (accurate to 0.000 1 g); place it in a 50 mL centrifuge tube; slowly add 20 mL of nitric acid solution (10+90). And ultrasonically treat it in an ultrasonic generator for 20 min (if there is turbidity, it can be heated to dissolve or 1.0 mL ~ 1.5 mL of hydrochloric acid can be added to dissolve); cool to room temperature; transfer to a 50 mL volumetric flask; and make constant volume with water. According to the labeled amount of minerals in the sample, dilute the sample solution appropriately with nitric acid solution (4+96); and mix well. Perform a blank test at the same time.

19.3 Instrument reference conditions

Optimize the instrument operating conditions so that the sensitivity and other indicators of the elements to be measured meet the analysis requirements; edit the determination method; select the appropriate analytical spectrum line of each element to be measured; and the instrument operating reference conditions and recommended analytical spectrum line of the elements to be measured are shown in Appendix D.

19.4 Drawing of standard curve

Inject the standard series working solution into the inductively coupled plasma emission spectrometer; measure the intensity signal response value of the analytical spectrum line of the elements to be measured in the standard working solution. And draw the standard curve with the concentration of the elements to be measured as the horizontal axis and the intensity signal response value of the analytical spectrum line as the vertical axis.

19.5 Determination of specimen solution

This is an excerpt of the PDF (Some pages are marked off intentionally)

Full-copy PDF can be purchased from 1 of 2 websites:

1. https://www.ChineseStandard.us

- SEARCH the standard ID, such as GB 4943.1-2022.
- Select your country (currency), for example: USA (USD); Germany (Euro).
- Full-copy of PDF (text-editable, true-PDF) can be downloaded in 9 seconds.
- Tax invoice can be downloaded in 9 seconds.
- Receiving emails in 9 seconds (with download links).

2. https://www.ChineseStandard.net

- SEARCH the standard ID, such as GB 4943.1-2022.
- Add to cart. Only accept USD (other currencies - https://www.ChineseStandard.us).
- Full-copy of PDF (text-editable, true-PDF) can be downloaded in 9 seconds.
- Receiving emails in 9 seconds (with PDFs attached, invoice and download links).

Translated by: Field Test Asia Pte. Ltd. (Incorporated & taxed in Singapore. Tax ID: 201302277C)

Contact: Wayne Zheng, Sales@ChineseStandard.net

Linkin: https://www.linkedin.com/in/waynezhengwenrui/

------ The End ------