GB

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National food safety standard –

Determination of multi-elements in foods

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National food safety standard – Determination of multi-elements in foods

1 Scope

This standard specifies inductively coupled plasma mass spectrometry (ICP-MS) and inductively coupled plasma emission spectrometry (ICP-OES) for the determination of multi-element in foods.

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

Method I: Inductively coupled plasma mass spectrometry (ICP-MS)

2 Principle

The sample is digested and determined by inductively coupled plasma mass spectrometry. It is subject to qualitative determination by the specific mass number of element (mass/charge ratio, m/z). The quantitative analysis is performed by the use of external standard method and by that the ratio between the strength ratio of the mass spectrometry signal of the tested element to the internal standard element mass spectrometry signal and the concentration of the element to be determined.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method are all guaranteed grade, the water is a level 1 water as specified in GB/T 6682.

3.1 Reagents

3.1.1 Nitric acid (HNO_3): Guaranteed grade or higher purity.

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

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

internal standard use solution of appropriate concentration, the concentration of the internal standard use solution is as shown in A.2.

Note: The internal standard solution can either be manually added quantitatively in the preparation of mixed standard working solution and sample digestion solution, or be added online by the instrument.

4 Instruments and equipment

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

4.2 Balance: The sensitivity is 0.1 mg and 1 mg.

4.3 Microwave digestion instrument: Equipped with PTFE digestion inner tank.

4.4 Pressure digestion tank: Equipped with PTFE digestion inner tank.

4.5 Constant temperature drying oven.

4.6 Temperature control electric heating plate.

4.7 Ultrasonic water bath.

4.8 Sample crushing equipment: Homogenizer, high-speed crusher.

5 Analytical procedures

5.1 Specimen preparation

5.1.1 Solid sample

5.1.1.1 Dry sample

For the samples of low moisture content such as beans, cereals, fungi, tea, dried fruits, baked goods, etc., TAKE the edible parts, if necessary, USE high-speed crusher to crush it evenly; for the uniform powder sample such as solid dairy products, protein powder, flour, etc., SHAKE it uniformly.

5.1.1.2 Fresh sample

For the samples of high moisture content such as vegetables, fruits, and aquatic products, it shall be washed clean when necessary, dried naturally, and the edible parts shall be homogenized uniformly; for such samples as meat, eggs, etc., the edible parts shall be homogenized uniformly.

5.1.1.3 Frozen and canned food

the digestion tank on the temperature control electric hot plate or in the ultrasonic bath, HEAT it at 100 °C for 30 min or PERFORM ultrasonic degassing for 2 min ~ 5 min, USE water to make the volume reach to 25 mL or 50 mL, MIX it uniformly to prepare for use, meanwhile PERFORM blank test.

5.3 Instrument reference conditions

5.3.1 Instrument operating conditions: Instrument operating conditions are shown in Table B.2; elemental analysis modes are shown in Table B.3.

Note: For instruments that do not have a suitable interference elimination mode, the interference correction equation shall be used to correct the measurement results. The interference correction equations for lead, cadmium, arsenic, molybdenum, selenium, vanadium, and other elements are given in Table B.4.

5.3.2 Determination reference conditions: After tuning the instrument to meet the measurement requirements, EDIT the measurement method and SELECT the appropriate internal standard element based on the nature of the element to be measured. SEE Table B.5 for m/z of the test element and the internal standard element.

5.4 Production of standard curves

INJECT the mixed standard solution into an inductively coupled plasma mass spectrometer, to measure the signal response values of the test element and the internal standard element. USE the concentration of the test element as the abscissa and the ratio of the response signal value of the test element to the selected internal standard element as the ordinate, to draw a standard curve.

5.5 Determination of specimen solution

Respectively INJECT the blank solution and the specimen solution into an inductively coupled plasma mass spectrometer, to measure the signal response values of the test element and the internal standard element, and to obtain the concentration of the test element in the digestion solution in accordance with the standard curve.

6 Expression of analysis results

6.1 Calculation of low-content test elements

The content of the low-content test element in the specimen is calculated in accordance with the formula (1):

$$X = \frac{(\rho - \rho_0) \times V \times f}{m \times 1\,000} \qquad \cdots\cdots (1)$$

12.2.1 Microwave digestion method

Same as 5.2.1.

12.2.2 Pressure tank digestion method

Same as 5.2.2.

12.2.3 Wet digestion method

Accurately WEIGH 0.5 g ~ 5 g (accurate to 0.001 g) or accurately PIPETTE 2.00 mL ~ 10.0 mL of specimen in glass or PTFE digestion vessel; for the sample containing ethanol or carbon dioxide, first HEAT it on a hot plate at low temperature to remove ethanol or carbon dioxide, ADD 10 mL of nitric acid-perchloric acid (10 + 1) mixed solution, MAKE it subject to digestion on an electric hot plate or graphite digestion device, if the digestion solution becomes brown and black in the digestion process, it may add a small amount of mixed acid appropriately, until white smoke is produced, the digestion solution is colorless, transparent or slightly yellow, COOL it, USE water to make its volume reach to 25 mL or 50 mL, MIX it uniformly to prepare for use; meanwhile PERFORM blank test.

12.2.4 Dry digestion method

Accurately WEIGH 1 g ~ 5 g (accurate to 0.01 g) or accurately PIPETTE 10.0 mL ~ 15.0 mL of specimen in the crucible, PLACE it in 500 °C ~ 550 °C muffle furnace to ash it for 5 h ~ 8 h, COOL it down. If the ash is not complete, there will be black charcoal particles, then ADD a little nitric acid to wet it after cooling, after drying it on an electric hot plate, TRANSFER it into the muffle furnace to continue ash it to white ash, COOL it down and TAKE it out, ADD 10 mL of nitric acid solution to dissolve it, USE water to make its volume reach to 25 mL or 50 mL, MIX it uniformly to prepare for use; meanwhile PERFORM blank test.

12.3 Instrument reference conditions

OPTIMIZE the operating conditions of the instrument, so that the sensitivity of the element to be determined meets the analysis requirements. EDIT the determination method and SELECT the appropriate analysis spectral line of each test element, the instrument operating conditions are as shown in B.3.1, the recommended analysis spectral line of the test element is as shown in Table B.6.

12.4 Production of standard curves

INJECT the standard series working solution into an inductively coupled plasma emission spectrometer, to measure the strength signal response value of the analytical spectral line of the test element. USE the concentration of the test