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NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 31604.49-2023

National Food Safety Standard - Food Contact Materials and Products - Determination of Multi-elements and Determination of Multi-element Migration

食品安全国家标准　食品接触材料及制品　多元素的测定和多元素迁移量的测定

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State Administration for Market Regulation.

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National Food Safety Standard - Food Contact Materials and Products - Determination of Multi-elements and Determination of Multi-element Migration

1 Scope

This Standard specifies the method for the determination of arsenic, cadmium, chromium and lead, and the determination of aluminum, arsenic, barium, cadmium, cobalt, chromium, copper, iron, lithium, manganese, molybdenum, nickel, lead, antimony, tin and zinc migration in food contact materials and products.

Part 1 is applicable to the determination of arsenic, cadmium, chromium and lead in food contact paper and cardboard materials and products, cork stoppers and bamboo and wood products.

Part 2 is applicable to the determination of aluminum, arsenic, barium, cadmium, cobalt, chromium, copper, iron, lithium, manganese, molybdenum, nickel, lead, antimony, tin and zinc migration in food contact plastic materials and products, food contact paints and coatings, food contact rubber materials and products, inks for food contact materials and products, adhesives for food contact materials and products, food contact paper and cardboard materials, pacifiers, enamel products, ceramic products, glass products, food contact plastic resins, food contact metal materials and products.

Part 1 - Determination of Arsenic, Cadmium, Chromium and Lead

Method 1 Inductively Coupled Plasma Mass Spectrometry

2 Principle

After the specimen is crushed, use nitric acid for digestion. After the obtained solution is diluted with water to a constant volume, adopt an inductively coupled plasma mass spectrometer to conduct the determination. Take the specific mass number of the element (mass-to-charge ratio, m/z) for qualitative analysis and the external standard method for quantitative analysis.

3 Reagents and Materials

Unless it is otherwise specified, the reagents used in this Method are all excellent-grade pure,

and the water is Grade-1 water specified in GB/T 6682.

3.1 Reagents

3.1.1 Nitric acid (HNO_3): excellent-grade pure or higher.

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

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

3.2 Preparation of Reagents

3.2.1 Nitric acid solution (5 + 95): measure-take 50 mL of nitric acid, slowly add it to 950 mL of water and evenly mix it.

3.2.2 Nitric acid solution (1 + 4): measure-take 1 L of nitric acid, slowly add it to 4 L of water and evenly mix it.

3.3 Reference Materials

3.3.1 Element standard solution (1,000 mg/L or 100 mg/L): for arsenic, cadmium, chromium and lead, adopt single-element or multi-element standard stock solutions certified by the state and awarded a reference material certificate. It shall remain valid for 1 year.

3.3.2 Internal standard element solution (1,000 mg/L or 100 mg/L): for scandium, germanium, rhodium, indium, rhenium and bismuth, adopt single-element or multi-element standard stock solutions certified by the state and awarded a reference material certificate. It shall remain valid for 2 years.

3.4 Preparation of Standard Solutions

3.4.1 Mixed standard working solution: accurately draw an appropriate amount of single-element or multi-element mixed standard stock solution, use nitric acid solution (5 + 95) to dilute it step by step to prepare a mixed standard series solution. The concentration of each element is shown in Table A.1 in Appendix A. After the mixed standard series solution is prepared, transfer it to a brown glass container and store it away from light at room temperature. It shall remain valid for 1 month.

NOTE: the concentration and range of the element in the standard series solution can be appropriately adjusted in accordance with the sensitivity and linear range of the instrument, and the actual content of each element in the specimen solution.

3.4.2 Internal standard working solution: take an appropriate amount of internal standard single-element stock solution or internal standard multi-element stock solution, use nitric acid solution (5 + 95) to prepare a multi-element internal standard working solution of an appropriate concentration. After the specimen solution is mixed, the reference concentrations of the internal standard elements are shown in A.5 in Appendix A. After the internal standard working solution is prepared, transfer it to a brown glass container and store it away from light at room

tank out of the digestion instrument. After the digestion tank has been completely cooled down, slowly open the inner cover. Use a small amount of water to rinse the inner cover twice and put it in the digestion tank. Place the digestion tank on the temperature-controllable electric hot plate and heat it at about 140 °C for 30 min, or place it in the ultrasonic cleaning machine for 5 min. Transfer all the digestion solution to a 25 mL or 50 mL volumetric flask, use water to reach a constant volume to the scale, evenly mix it and reserve it for later testing.

5.2.1.2 Blank test

Except that no specimen is added, proceed in accordance with 5.2.1.1 to obtain a blank test solution.

5.2.2 Pressure tank digestion method

5.2.2.1 Preparation of specimen solution

Weigh-take 0.5 g (accurate to 0.1 mg) of the crushed specimen, place it in the polytetrafluoroethylene digestion inner tank, add 5 mL ~ 8 mL of nitric acid, cover it and leave it for 1 h. Seal the digestion inner tank in the stainless-steel outer tank, and place it in a constant-temperature drying oven for digestion (see Table B.1 in Appendix B for the reference conditions of digestion). After digestion, wait until the digestion tank has been completely cooled down before slowly opening the inner cover. Use a small amount of water to rinse the inner cover twice and put it in the digestion tank. Place the digestion tank on the temperature-controllable electric hot plate and heat it at about 140 °C for 30 min, or place it in the ultrasonic cleaning machine for 5 min. Transfer all the digestion solution to a 25 mL or 50 mL volumetric flask, use water to reach a constant volume to the scale, evenly mix it and reserve it for later testing.

5.2.2.2 Blank test

Except that no specimen is added, proceed in accordance with 5.2.2.1 to obtain a blank test solution.

5.3 Reference Conditions of Instrument

5.3.1 Instrument operating conditions

The reference working conditions of the instrument are shown in Table B.2 in Appendix B. The element reference analysis mode is shown in Table B.3 in Appendix B.

NOTE: for instruments that do not have a suitable interference elimination mode, it is necessary to adopt the interference correction equations to correct the determination results. The interference correction equation for arsenic, cadmium and lead, etc. are shown in Table B.4 in Appendix B.

5.3.2 Reference conditions of determination

Under the operating conditions of the selected instrument, edit the determination method; in accordance with the properties of the element to be determined, select the corresponding

10 Reagents and Materials

Unless it is otherwise specified, the reagents used in this Method are all excellent-grade pure, and the water is Grade-1 water specified in GB/T 6682.

10.1 Reagents

10.1.1 Nitric acid (HNO_3): excellent-grade pure or higher.

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

10.2 Preparation of Reagents

10.2.1 Nitric acid solution (5 + 95): measure-take 50 mL of nitric acid, slowly add it to 950 mL of water and evenly mix it.

10.2.2 Nitric acid solution (1 + 4): measure-take 1 L of nitric acid, slowly add it to 4 L of water and evenly mix it.

10.3 Reference Materials

Element standard solution (1,000 mg/L or 100 mg/L): for arsenic, cadmium, chromium and lead, adopt single-element or multi-element standard stock solutions certified by the state and awarded a reference material certificate. It shall remain valid for 1 year.

10.4 Preparation of Standard Solution

Mixed standard working solution: accurately draw an appropriate amount of single-element or multi-element mixed standard stock solution, use nitric acid solution (5 + 95) to dilute it step by step to prepare a mixed standard series solution. The mass concentration of each element is shown in Table A.2 in Appendix A. After the mixed standard series solution is prepared, transfer it to a brown glass container and store it away from light at room temperature. It shall remain valid for 1 month.

NOTE: the concentration and range of the element in the standard series solution can be appropriately adjusted in accordance with the sensitivity and linear range of the instrument, and the actual content of each element in the specimen solution.

11 Instruments and Equipment

NOTE: all glassware and plasticware need to be soaked in nitric acid solution (1 + 4) overnight, rinsed with water and reserved for later use.

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

11.2 Balance: with a division value of 0.1 mg.

17.2.1 Food simulant: 4% (volume fraction) acetic acid, 10% (volume fraction) ethanol, 20% (volume fraction) ethanol, 50% (volume fraction) ethanol, 95% (volume fraction) ethanol, 1 g/L citric acid, 5 g/L citric acid and artificial tap water, which are prepared in accordance with the stipulations of GB 5009.156.

17.2.2 Nitric acid solution (5 + 95): measure-take 50 mL of nitric acid, slowly add it to 950 mL of water and evenly mix it.

17.2.3 Nitric acid solution (1 + 4): measure-take 1 L of nitric acid, slowly add it to 4 L of water and evenly mix it.

17.3 Reference Materials

17.3.1 Element standard solution (1,000 mg/L or 100 mg/L): for aluminum, arsenic, barium, cadmium, cobalt, chromium, copper, iron, lithium, manganese, molybdenum, nickel, lead, antimony, tin and zinc, adopt single-element or multi-element standard stock solutions certified by the state and awarded a reference material certificate. It shall remain valid for 1 year.

17.3.2 Internal standard element solution (1,000 mg/L or 100 mg/L): for scandium, germanium, rhodium, indium, rhenium and bismuth, adopt single-element or multi-element standard stock solutions certified by the state and awarded a reference material certificate. It shall remain valid for 2 years.

17.4 Preparation of Standard Solutions

17.4.1 Mixed standard working solution: accurately draw an appropriate amount of single-element or multi-element mixed standard stock solution, use 4% (volume fraction) acetic acid solution [applicable to 4% (volume fraction) acetic acid specimen solution], 1 g/L citric acid solution and 5 g/L citric acid solution (respectively applicable to citric acid specimen solutions of different concentrations), nitric acid solution (5 + 95) (applicable to artificial tap water, alcohol, isooctane and olive oil specimen solutions) to dilute it step by step to prepare a mixed standard series solution. The concentration of each element is shown in Table A.3 in Appendix A. After the mixed standard series solution is prepared, transfer it to a brown glass container and store it at room temperature. It shall remain valid for 1 month.

NOTE: the concentration and range of the element in the standard series solution can be appropriately adjusted in accordance with the sensitivity and linear range of the instrument, and the actual content of each element in the food simulant migration specimen solution.

17.4.2 Internal standard working solution: take an appropriate amount of internal standard single-element stock solution or internal standard multi-element stock solution, use nitric acid solution (5 + 95) to prepare a multi-element internal standard used solution of an appropriate concentration. After the specimen solution is mixed, the reference concentrations of the internal standard elements are shown in A.5 in Appendix A. After the internal standard working solution is prepared, transfer it to a brown glass container and store it at room temperature. It shall remain valid for 1 month.

NOTE: the internal standard solution can be manually and quantitatively added when preparing the mixed standard series solution and the specimen solution to be tested, or it can be added online by the instrument. The internal standard concentration can be adjusted in accordance with the ratio of the internal standard injection volume and the sample injection volume.

18 Instruments and Equipment

NOTE: all glassware and plasticware need to be soaked in nitric acid solution (1 + 4) overnight, rinsed with water and reserved for later use.

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

18.2 Analytical balance: with a division value of 0.01 g and 0.1 mg, respectively.

18.3 Temperature-controllable electric hot plate.

18.4 Rotary evaporator.

18.5 Microporous filter membrane: hydrophilic PTFE, 0.45 μm.

19 Analytical Procedures

19.1 Specimen Preparation

19.1.1 Types of food simulants

In this Standard, the migration test adopts 4% (volume fraction) acetic acid, 10% (volume fraction) ethanol, 20% (volume fraction) ethanol, 50% (volume fraction) ethanol, 95% (volume fraction) ethanol, 1 g/L citric acid, 5 g/L citric acid, artificial tap water, olive oil and isooctane as food simulants.

19.1.2 Migration test

In accordance with the intended purposes and usage conditions of the sample to be tested, and the migration test methods and test conditions specified in GB 5009.156, GB 31604.1 and the stipulations of relevant product standards, conduct the migration test. After the food simulant soaking solution is thoroughly mixed, take part of the soaking solution for analysis.

19.1.3 Preparation of specimen solution

19.1.3.1 4% (volume fraction) acetic acid and citric acid specimen solution

Filter 4% (volume fraction) acetic acid soaking solution, 1 g/L citric acid and 5 g/L citric acid soaking solution through the microporous filter membrane, then, directly inject it for determination.

19.1.3.2 Artificial tap water specimen solution

Take 2.5 mL of nitric acid and place it in a 50 mL volumetric flask. Use the filtrate of the artificial tap water soaking solution filtered through the microporous filter membrane to reach a constant volume, evenly mix it and reserve it for later use.

19.1.3.3 Alcohol specimen solution

Accurately weigh-take 10 g (accurate to 0.01 g) of 10% (volume fraction) ethanol, 20% (volume fraction) ethanol, 50% (volume fraction) ethanol and 95% (volume fraction) ethanol soaking solution into a digestion tube or 100 mL conical flask. Place it on a temperature-controllable electric hot plate, at about 150 °C, evaporate the specimen solution to almost dryness, then, add 5 mL of nitric acid solution (5 + 95), continue heating for 5 min ~ 10 min and prevent evaporation to dryness. Take it out and cool to room temperature, then, use nitric acid solution (5 + 95) to transfer and reach a constant volume of 10 mL, evenly mix it and reserve it for later use.

19.1.3.4 Olive oil specimen solution

Weigh-take 0.5 g (accurate to 0.1 mg) of olive oil soaking solution, in accordance with the stipulations of 5.2, conduct pre-treatment.

19.1.3.5 Isooctane specimen solution

Accurately weigh-take 10 g (accurate to 0.01 g) of isooctane soaking solution into a 50 mL pear-shaped distillation bottle. In a normal-temperature water bath, conduct rotary evaporation under reduced pressure to dryness, then, use nitric acid (5 + 95) to rinse the pear-shaped distillation bottle for three times, combine the washing solution and use nitric acid solution (5 + 95) to reach a constant volume of 10 mL, evenly mix it and reserve it for later testing.

19.1.3.6 Blank test solution

Food simulants that are not in contact with food contact materials and products are treated in accordance with 19.1.2 and 19.1.3 to obtain a blank test solution.

19.2 Reference Conditions of Instrument

Same as 5.3.

19.3 Drawing of Standard Curve

In accordance with the concentration from low to high, determine the signal response values of the element to be determined and the internal standard element in the mixed standard working solution containing the internal standard element. Take the concentration of the element to be determined as the x-coordinate, and the ratio of the signal response value of the element to be determined to the signal response value of the selected internal standard element as the y-coordinate to draw a standard curve.

19.4 Determination of Specimen Solution

24 Reagents and Materials

Unless it is otherwise specified, the reagents used in this Method are all excellent-grade pure, and the water is Grade-1 water specified in GB/T 6682.

24.1 Reagents

24.1.1 Nitric acid (HNO_3): excellent-grade pure or higher.

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

24.1.3 Reagents required for the preparation of food simulants: in accordance with the stipulations of GB 5009.156.

24.2 Preparation of Reagents

24.2.1 Food simulant: 4% (volume fraction) acetic acid, 10% (volume fraction) ethanol, 20% (volume fraction) ethanol, 50% (volume fraction) ethanol, 95% (volume fraction) ethanol, 1 g/L citric acid, 5 g/L citric acid and artificial tap water, which are prepared in accordance with the stipulations of GB 5009.156.

24.2.2 Nitric acid solution (5 + 95): measure-take 50 mL of nitric acid, slowly add it to 950 mL of water and evenly mix it.

24.2.3 Nitric acid solution (1 + 4): measure-take 1 L of nitric acid, slowly add it to 4 L of water and evenly mix it.

24.3 Reference Materials

Element standard solution (1,000 mg/L or 100 mg/L): for aluminum, arsenic, barium, cadmium, cobalt, chromium, copper, iron, lithium, manganese, molybdenum, nickel, lead, antimony, tin and zinc, adopt single-element or multi-element standard stock solutions certified by the state and awarded a reference material certificate. It shall remain valid for 1 year.

24.4 Preparation of Standard Solution

Mixed standard working solution: accurately draw an appropriate amount of single-element or multi-element mixed standard stock solution, use 4% (volume fraction) acetic acid solution [applicable to 4% (volume fraction) acetic acid specimen solution], 1 g/L citric acid solution and 5 g/L citric acid solution (respectively applicable to citric acid specimen solutions of different concentrations), nitric acid solution (5 + 95) (applicable to artificial tap water, alcohol and isooctane specimen solutions) to dilute it step by step to prepare a mixed standard series solution. The concentration of each element is shown in Table A.4 in Appendix A. After the mixed standard series solution is prepared, transfer it to a brown glass container and store it at room temperature. It shall remain valid for 1 month.

NOTE: the concentration and range of the element in the standard series solution can be

appropriately adjusted in accordance with the sensitivity and linear range of the instrument, and the actual content of each element in the food simulant migration specimen solution.

25 Instruments and Equipment

NOTE: all glassware and plasticware need to be soaked in nitric acid solution (1 + 4) overnight, rinsed with water and reserved for later use.

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

25.2 Analytical balance: with a division value of 0.01 g and 0.1 mg, respectively.

25.3 Temperature-controllable electric hot plate.

25.4 Rotary evaporator.

25.5 Microporous filter membrane: hydrophilic PTFE, 0.45 μm.

26 Analytical Procedures

26.1 Specimen Preparation

The preparation of 4% (volume fraction) acetic acid, 10% (volume fraction) ethanol, 20% (volume fraction) ethanol, 50% (volume fraction) ethanol, 95% (volume fraction) ethanol, 1 g/L citric acid, 5 g/L citric acid, artificial tap water and isooctane specimen solution is the same as 19.1.

26.2 Reference Conditions of Instrument

Under the working conditions of the selected instrument, make the indicators, for example, sensitivity, of the elements to be determined reach the analysis requirements. Edit the determination method and select the appropriate analytical spectral line for each element to be determined. The reference conditions of instrument operation are shown in B.3.1 in Appendix B. The recommended analytical spectral lines of the elements to be determined are shown in Table B.6 in Appendix B.

26.3 Drawing of Standard Curve

Inject the mixed standard working solution into the inductively coupled plasma optimal emission spectrometer. In accordance with the concentration from low to high, determine the signal response values of the intensity of the analytical spectral lines of the elements to be determined. Take the concentration of the element to be determined as the x-coordinate, and the intensity response value of its analytical spectral line as the y-coordinate to draw a standard curve.

26.4 Determination of Specimen Solution

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