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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 5009.15-2023

**National Food Safety Standard - Determination of Cadmium
in Foods**

食品安全国家标准 食品中镉的测定

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National Food Safety Standard - Determination of Cadmium in Foods

1 Scope

This Standard specifies the method for the determination of cadmium in foods by graphite furnace atomic absorption spectrometry and inductively coupled plasma mass spectrometry.

This Standard is applicable to the determination of cadmium in foods.

Method I - Graphite Furnace Atomic Absorption Spectrometry

2 Principle

After the specimen is digested, atomize it in a graphite furnace, at 228.8 nm, determine the absorbance. Within a certain concentration range, the absorbance value of cadmium is proportional to the cadmium content. Conduct quantitative comparison with standard series solutions.

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-2 water specified in GB/T 6682.

3.1 Reagents

3.1.1 Nitric acid (HNO_3).

3.1.2 Perchloric acid (HClO_4).

3.1.3 Ammonium dihydrogen phosphate ($\text{NH}_4\text{H}_2\text{PO}_4$).

3.1.4 Palladium nitrate [$\text{Pd}(\text{NO}_3)_2$].

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 + 9): measure-take 50 mL of nitric acid, slowly add it to 450 mL of

water and evenly mix it.

3.2.3 Ammonium dihydrogen phosphate - palladium nitrate mixed solution: weigh-take 0.02 g of palladium nitrate, add a small amount of nitric acid solution (1 + 9) to dissolve it, then, add 2 g of ammonium dihydrogen phosphate. After dissolving, use nitric acid solution (5 + 95) to reach a constant volume of 100 mL, and evenly mix it.

3.3 Reference Material

Cadmium chloride ($\text{CdCl}_2 \bullet 2.5\text{H}_2\text{O}$, CAS No.: 7790-78-5): purity > 99.99%, or a standard substance certified by the state and awarded a reference material certificate.

3.4 Preparation of Standard Solutions

3.4.1 Cadmium standard stock solution (100 mg/L): accurately weigh-take 0.2032 g of cadmium chloride, use a small amount of nitric acid solution (1 + 9) to dissolve it, transfer it into a 1,000 mL volumetric flask. Add water to the scale and evenly mix it. The mass concentration of cadmium in this solution is 100 mg/L.

3.4.2 Cadmium standard intermediate solution (100 $\mu\text{g/L}$): accurately draw 1.00 mL of cadmium standard stock solution (100 mg/L) into a 10 mL volumetric flask, add nitric acid solution (5 + 95) to the scale, and evenly mix it. Then, accurately draw 1.00 mL of the above-mentioned solution into a 100 mL volumetric flask, add nitric acid solution (5 + 95) to the scale, and evenly mix it. The mass concentration of cadmium in this solution is 100 $\mu\text{g/L}$.

3.4.3 Cadmium standard series of working solutions: respectively and accurately draw 0 mL, 0.200 mL, 0.500 mL, 1.00 mL, 2.00 mL and 4.00 mL of cadmium standard intermediate solution (100 $\mu\text{g/L}$) into a 100 mL volumetric flask. Add nitric acid solution (5 + 95) to the scale, and evenly mix it. The mass concentration of cadmium in this series of solutions is 0 $\mu\text{g/L}$, 0.200 $\mu\text{g/L}$, 0.500 $\mu\text{g/L}$, 1.00 $\mu\text{g/L}$, 2.00 $\mu\text{g/L}$ and 4.00 $\mu\text{g/L}$. Prepare them right before use.

NOTE: the mass concentration of cadmium in the standard series of solutions can be determined in accordance with the sensitivity of the instrument and the actual cadmium content in the sample.

4 Instruments and Equipment

NOTE: all glassware and polytetrafluoroethylene digestion inner tanks need to be soaked in nitric acid solution (1 + 5) overnight, repeatedly rinsed with tap water, and finally, thoroughly rinsed with water.

4.1 Atomic absorption spectrometer: equipped with graphite furnace atomizer and cadmium hollow cathode lamp.

4.2 Electronic balance: with a division value of 0.1 mg and 1 mg.

accurately transfer-take or weigh-take 0.500 mL (g) ~ 5.00 mL (g) (accurate to 0.001 g) into a digestion tube with scale. For samples containing ethanol or carbon dioxide, firstly heat them at a low temperature to remove ethanol or carbon dioxide, add 10 mL of nitric acid and 0.5 mL of perchloric acid, and digest on an adjustable electric furnace (reference conditions: at 120 °C, maintain for 0.5 h ~ 1 h, raise to 180 °C, maintain for 2 h ~ 4 h, raise to 200 °C ~ 220 °C). If the digestion solution is brown, after cooling, add a small amount of nitric acid and digest it, until it emits white smoke, and the digestion solution turns colorless, transparent or slightly yellow. Drive acid to about 1 mL, take out the digestion tube, cool it and use water to reach a constant volume of 10 mL or 25 mL, evenly mix it and reserve it for later use. Meanwhile, conduct a blank test. Alternatively, a conical flask can also be used; on an adjustable electric heating plate, in accordance with the above-mentioned operation method, perform wet digestion.

5.2.1.2 Microwave digestion method

For solid specimens, weigh-take 0.2 g ~ 0.5 g (accurate to 0.001 g; for samples containing relatively high-water content, the sampling size can be appropriately increased to 1 g). For liquid specimens, accurately transfer-take or weigh-take 0.500 mL (g) ~ 3.00 mL (g) (accurate to 0.001 g) into a microwave digestion tank. For samples containing ethanol or carbon dioxide, firstly heat them at a low temperature to remove ethanol or carbon dioxide, add 5 mL ~ 10 mL of nitric acid, in accordance with the operation procedures of microwave digestion, digest the specimen. For the digestion conditions, refer to Table A.1 in Appendix A. When necessary, after adding acid, cover and let it stand for 1 h or overnight, then, in accordance with the operation procedures of microwave digestion, digest the specimen. After cooling, take out the digestion tank; at 140 °C ~ 160 °C, drive acid to about 1 mL. After the digestion tank cools, transfer the digestion solution into a 10 mL or 25 mL volumetric flask. Use a small amount of water to wash the digestion tank 2 ~ 3 times, combine the washing liquid in the volumetric flask and use water to reach a constant volume to the scale; evenly mix it and reserve it for later use. Meanwhile, conduct a blank test.

5.2.1.3 Pressure tank digestion method

For solid specimens, weigh-take 0.2 g ~ 1 g (accurate to 0.001 g; for samples containing relatively high-water content, the sampling size can be appropriately increased to 2 g). For liquid specimens, accurately transfer-take or weigh-take 0.500 mL (g) ~ 5.00 mL (g) (accurate to 0.001 g) into a digestion inner tank. For samples containing ethanol or carbon dioxide, firstly heat them at a low temperature to remove ethanol or carbon dioxide and add 5 mL ~ 10 mL of nitric acid. Properly put on the inner cover, tighten the stainless-steel jacket, put it into a constant-temperature drying oven; at 140 °C ~ 160 °C, maintain for 4 h ~ 5 h. When necessary, after adding acid, cover and let it stand for 1 h or overnight, then, tighten the stainless-steel jacket and put it into a constant-temperature drying oven to digest the specimen. After cooling, slowly unscrew the stainless-steel jacket, take out the digestion inner tank; at 140 °C ~ 160 °C, drive acid to about 1 mL. After cooling, transfer the digestion solution to a 10 mL or 25 mL volumetric flask, use a small amount of water to wash the inner tank and inner cover for 2 ~ 3 times, combine the washing liquid in the volumetric flask and use water to reach a constant volume to the scale; evenly mix it and reserve it for later use. Meanwhile, conduct a blank test.

5.3 Reference Conditions of Instruments

See Table A.2 for instrument operating conditions.

5.4 Drawing of Standard Curve

In accordance with the mass concentration from low to high, respectively take 10 µL of the standard series of solutions and 5 µL of the ammonium dihydrogen phosphate - palladium nitrate mixed solution (select the optimal sample injection volume in accordance with the instrument used), meanwhile, inject it into the graphite tube. After atomization, determine the absorbance value, and by taking the mass concentration as the x-coordinate and the absorbance value as the y-coordinate, draw a standard curve.

5.5 Determination of Specimen Solution

Under the same test conditions as the standard curve, draw 10 µL of blank solution or specimen digestion solution, and 5 µL of the ammonium dihydrogen phosphate - palladium nitrate mixed solution (select the optimal sample injection volume in accordance with the instrument used), meanwhile, inject it into the graphite tube. After atomization, determine the absorbance value. In accordance with the standard curve, obtain the mass concentration of cadmium in the solution to be tested. If the determination result is beyond the range of the standard curve, use nitric acid solution (5 + 95) to dilute it, then, perform the determination.

6 Expression of Analysis Results

The content of cadmium in the specimen is calculated in accordance with Formula (1).

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

Where,

X ---the cadmium content in the specimen, expressed in (mg/kg) or (mg/L);

ρ ---the mass concentration of cadmium in the specimen digestion solution, expressed in (µg/L);

ρ_0 ---the mass concentration of cadmium in the blank solution, expressed in (µg/L);

f ---dilution factor;

V ---the constant volume of the specimen digestion solution, expressed in (mL);

m ---the mass or volume of the specimen, expressed in (g) or (mL);

1,000---conversion factor.

When the cadmium content is ≥ 0.1 mg/kg (mg/L), the calculation results shall retain 3 significant figures. When the cadmium content is < 0.1 mg/kg (mg/L), the calculation results

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