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National food safety standard - Determination of nickel in food

食品安全国家标准 食品中镍的测定

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1 Scope

This Standard specifies the methods for the determination of nickel in food by graphite furnace atomic absorption spectrometry, inductively coupled plasma mass spectrometry and inductively coupled plasma emission spectrometry.

This Standard applies to the determination of nickel in food.

Method I – Graphite furnace atomic absorption spectrometry

2 Principle

After digestion treatment, the sample is atomized in a graphite furnace and the absorbance is measured at 232.0 nm. Within a certain concentration range, the absorbance value of nickel is proportional to the nickel content, and the external standard method is used for quantification.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method are guaranteed reagents, and the water is grade-II water as 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 Palladium nitrate [$Pd(NO_3)_2$].

3.1.4 Ammonium dihydrogen phosphate ($NH_4H_2PO_4$).

3.2 Preparation of reagents

3.2.1 Nitric acid solution (5+95): Measure 50 mL of nitric acid; slowly add it to 950 mL of water; mix well.

3.2.2 Nitric acid solution (1+1): Measure 500 mL of nitric acid; slowly add it to 500 mL of water; mix well.

3.2.3 Nitric acid solution (1+5): Measure 100 mL of nitric acid; slowly add it to 500 mL of water; mix well.

3.2.4 Ammonium dihydrogen phosphate-palladium nitrate solution: Weigh 0.02 g of palladium nitrate; add a small amount of nitric acid solution (1+1) to dissolve it; then, add 2 g of ammonium dihydrogen phosphate; after dissolution, use nitric acid solution (1+1) to adjust the volume to 100 mL; mix well.

3.3 Standards

Metallic nickel (Ni, CAS number: 7440-02-0): purity >99.99%, or nickel standard solution certified by the country and awarded a standard material certificate.

3.4 Preparation of standard solutions

3.4.1 Nickel standard stock solution (1 000 mg/L): Accurately weigh 1 g (accurate to 0.0001 g) of metallic nickel; add 30 mL of nitric acid solution (1+1); heat to dissolve; transfer it to 1000 mL volumetric flask; add water to dilute to the mark; mix well. Refrigerate this solution at 0 ℃ ~ 5 ℃; it is valid for 6 months.

3.4.2 Nickel standard intermediate solution (10.0 mg/L): Accurately draw 1.00 mL of nickel standard stock solution (1 000 mg/L) into a 100 mL volumetric flask; use nitric acid solution (5+95) to dilute it to the mark; mix well. Refrigerate this solution at 0 ℃ ~ 5 ℃; it is valid for 1 month.

3.4.3 Nickel standard solution (1.00 mg/L): Accurately draw 10.00 mL of nickel standard intermediate solution (10.0 mg/L) into a 100 mL volumetric flask; use nitric acid solution (5+95) to adjust the volume to the mark; mix well. Refrigerate this solution at 0 ℃ ~ 5 ℃; it is valid for 1 month.

3.4.4 Nickel standard series solution: Respectively take 0 mL, 0.25 mL, 0.50 mL, 1.00 mL, 2.00 mL and 2.50 mL of nickel standard solution (1.00 mg/L) into the 100 mL volumetric flasks; add nitric acid solution (5+95) to the mark; mix well. The mass concentrations of this nickel standard series solution are 0 μg/L, 2.50 μg/L, 5.0 μg/L, 10.0 μg/L, 20.0 μg/L and 25.0 μg/L, respectively. Prepare when necessary.

Note: The mass concentration of nickel in the standard series solution can be determined based on the sensitivity of the instrument, the actual nickel content in the sample, and different instrument models.

4 Instruments and apparatuses

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

Mix well.

5.2 Sample digestion

5.2.1 Wet digestion method

Weigh 0.5 g ~ 2 g (accurate to 0.001 g) of the solid sample or accurately transfer 0.50 mL ~ 3.00 mL of the liquid sample into a graduated digestion tube. Heat samples containing ethanol or carbon dioxide first at low temperature on an electric hot plate to remove the ethanol or carbon dioxide. Add 10 mL of nitric acid and 0.5 mL of perchloric acid; put a few glass beads; digest it on an adjustable electric furnace (reference conditions: 120 °C for 0.5 h ~ 1 h; rise to 180 °C for 2 h ~ 4 h, rise to 200 °C ~ 220 °C). If the digestive juice is brown, add a small amount of nitric acid and digest until white smoke is emitted and the digestive juice is colorless, transparent or slightly yellow. Remove acid to nearly 1 mL; stop digestion; cool and add water to dilute to 10 mL or 25 mL; mix well and set aside. Do a reagent blank test at the same time. Alternatively, use an Erlenmeyer flask and perform wet digestion on an adjustable electric heating plate according to the above operation methods.

5.2.2 Microwave digestion

Weigh 0.2 g ~ 0.5 g (accurate to 0.001 g) of the solid sample or accurately transfer 0.50 mL ~ 2.00 mL of the liquid sample into the microwave digestion tank. For samples containing ethanol or carbon dioxide, first heat at low temperature on an electric hot plate to remove the ethanol or carbon dioxide; add 5 mL ~ 10 mL of nitric acid; digest the sample according to the operating steps of microwave digestion. For digestion conditions, refer to Table A.1 of Appendix A. After cooling, take out the digestion tank; reduce the acid to about 1 mL at 140 °C ~ 160 °C on the electric heating plate. After the digestion tank cools off, transfer the digestive juice to a 10 mL or 25 mL volumetric flask; use a small amount of water to wash the digestion tank 2 ~ 3 times; combine the cleaning mixture in the volumetric flask and use water to adjust the volume to the mark; mix well and set aside. Do a reagent blank test at the same time.

5.2.3 Pressure tank digestion

Weigh 0.2 g ~ 1 g (accurate to 0.001 g) of the solid sample or accurately transfer 0.50 mL ~ 5.00 mL of the liquid sample into the digestion inner tank. For samples containing ethanol or carbon dioxide, first heat at low temperature on an electric hot plate to remove the ethanol or carbon dioxide; add 5 mL of nitric acid. Close the inner cover; screw the stainless-steel jacket tightly; put it in a constant-temperature drying oven; keep it at 140 °C ~ 160 °C for 4 h ~ 5 h. After cooling, slowly unscrew the outer tank, take out the digestion inner tank, and place it on an adjustable electric hot plate to reduce acid to about 1 mL at 140 °C ~ 160 °C. 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 2 ~ 3 times; combine the cleaning mixture in the volumetric flask; use

Where:

X – the nickel content in the sample, in milligrams per kilogram (mg/kg) or milligrams per liter (mg/L);

ρ – the mass concentration of nickel in the sample solution, in micrograms per liter (μg/L);

f – dilution factor;

ρ_0 – the mass concentration of nickel in the blank solution, in micrograms per liter (μg/L);

V – the constant volume of the sample digestion solution, in milliliters (mL);

m – the weighing amount or pipetting volume of the sample, in grams (g) or milliliters (mL);

1 000 – Conversion factor.

When the nickel content is ≥1.00 mg/kg (or mg/L), retain 3 significant figures for the calculation result; when the nickel content is <1.00 mg/kg (or mg/L), retain 2 significant figures for the calculation result.

7 Precision

When the nickel content in the sample is greater than 1 mg/kg (or mg/L), the absolute difference between the two independent measurement results obtained under repeatability conditions shall not exceed 10% of the arithmetic mean; when it is less than or equal to 1 mg/kg (or mg/L) and greater than 0.1 mg/kg (or mg/L), the absolute difference between two independent determination results obtained under repeatability conditions shall not exceed 15% of the arithmetic mean; when it is less than or equal to 0.1 mg/kg (or mg/L), 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 0.5 g (or 0.5 mL) and the constant volume is 10 mL, the detection limit of the method is 0.02 mg/kg (or 0.02 mg/L), and the quantitation limit is 0.05 mg/kg (or 0.05 mg/ L).

Method II – Inductively coupled plasma mass spectrometry

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