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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 5009.123-2014

**National Food Safety Standard - Determination of
chromium in foods**

食品安全国家标准 食品中铬的测定

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National Food Safety Standard - Determination of chromium in foods

1 Scope

This Standard specifies the graphite furnace atomic absorption spectrometry method for the determination of chromium in foods.

This Standard applies to the determination of chromium in various foods.

2 Principle

After the sample is digested, use the graphite furnace atomic absorption spectrometry to measure the absorption value at 357.9 nm; compare the absorption value with the standard series solution within a certain concentration range.

3 Reagents and materials

Note: Unless otherwise specified, the reagents which are used in this method are all guaranteed reagents; the water is grade-II water that is 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 ($\text{NH}_4\text{H}_2\text{PO}_4$).

3.2 Preparation of reagents

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

3.2.2 Nitric acid solution (1+1): Measure 250 mL of nitric acid and slowly pour it into 250 mL of water; mix well.

3.2.3 Ammonium dihydrogen phosphate solution (20 g/L): Weigh 2.0 g of ammonium dihydrogen phosphate; dissolve in water; dilute to 100 mL; mix well.

5 Analysis steps

5.1 Pretreatment of the sample

5.1.1 For grains, beans, after removing the impurities, crush them and put them into clean containers as samples. Seal and mark; the samples shall be stored at room temperature.

5.1.2 For fresh samples of high moisture content, such as vegetables, fruits, fish, meat and eggs, directly stir them into homogenate and put them into clean containers as samples. Seal and mark. The samples shall be kept in the refrigerator freezer.

5.2 Sample digestion

5.2.1 Microwave digestion

Accurately weigh 0.2 g ~ 0.6 g (accurate to 0.001 g) of the sample in the microwave digestion tank; add 5 mL of nitric acid; digest the sample according to the microwave digestion steps (see A.1 for digestion conditions). After cooling, take out the digestion tank; reduce the acid to 0.5 mL ~ 1.0 mL at 140 °C ~ 160 °C on the electric heating plate. After the digestion tank is left cold, transfer the digestion solution to a 10 mL volumetric flask; use a small amount of water to wash the digestion tank 2 ~ 3 times; combine the washing solution; use water to dilute to the mark. Do a reagent blank test at the same time.

5.2.2 Wet digestion

Accurately weigh 0.5 g ~ 3 g of the sample (accurate to 0.001 g) into the digestion tube; add 10 mL of nitric acid, 0.5 mL of perchloric acid; digest on an adjustable electric furnace (reference conditions: keep at 120 °C for 0.5 h ~ 1 h; heat up to 180 °C for 2 h ~ 4 h; heat up to 200 °C ~ 220 °C). If the digestive juice is brown, add more nitric acid to dissolve until white smoke appears; when the digestive juice is colorless, transparent or slightly yellow, take out the digestive tube and use water to dilute to 10 mL after cooling. Do a reagent blank test at the same time.

5.2.3 High pressure digestion

Accurately weigh 0.3 g ~ 1 g of sample (accurate to 0.001 g) into the digestion inner tank; 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. Naturally cool in the box to room temperature; slowly unscrew the outer tank; take out the digestion inner tank; place it on the adjustable electric heating plate at 140 °C ~ 160 °C to reduce the acid to 0.5 mL ~ 1.0 mL. After cooling, transfer the digestion solution to a 10 mL volumetric

$$X = \frac{(c - c_0) \times V}{m \times 1\,000} \dots\dots\dots (1)$$

Where:

X -- content of chromium in the sample, in milligrams per kilogram (mg/kg);

c -- content of chromium in the working sample solution, in nanograms per milliliter (ng/mL);

c₀ -- content of chromium in the blank solution, in nanograms per milliliter (ng/mL);

V -- total volume of the sample digestion solution at a constant volume, in milliliters (mL);

m -- sample weighing amount, in grams (g);

1 000 -- conversion coefficient.

When the analysis result is ≥1 mg/kg, retain three significant digits; when the analysis result is <1 mg/kg, retain two significant digits.

7 Precision

The absolute difference of two independent test results under repeatability cannot exceed 20% of the arithmetic mean value.

8 Others

Calculate by the weighing amount of 0.5 g and the constant volume of 10 mL; the detection-limit of the method is 0.01 mg/kg; the quantitation-limit is 0.03 mg/kg.

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