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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 5009.269-2016

National Food Safety Standard - Determination of Talcum Powder in Food

食品安全国家标准

食品中滑石粉的测定

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People's Republic of China;**

China Food and Drug Administration.

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National Food Safety Standard -

Determination of Talcum Powder in Food

1 Scope

This Standard specifies the method of atomic absorption spectrometry in the determination of talcum powder in food.

This Standard is applicable to the determination of talcum powder in food.

2 Principle

The main component of talcum powder is natural hydrated magnesium silicate ($3\text{MgO} \cdot 4\text{SiO}_2 \cdot \text{H}_2\text{O}$). Use nitric acid + perchloric acid (or nitric acid + hydrogen peroxide) to digest the sample; filter it; separate talcum powder from other magnesium-containing substances. Talcum powder reacts with hydrofluoric acid, generates magnesium salt that is soluble in water. Use the method of atomic absorption spectrometry to determine magnesium content; calculate the content of talcum powder in accordance with magnesium content.

3 Reagents and Materials

Warning---hydrofluoric acid is highly toxic. Pay attention to safety protection. Avoid skin exposure or inhalation.

Unless it is otherwise stipulated, reagents that are used in this method are all analytical purity; use Grade-2 water stipulated in GB/T 6682.

3.1 Reagents

3.1.1 Nitric acid (HNO_3).

3.1.2 Hydrochloric acid (HCl).

3.1.3 Perchloric acid (HClO_4).

3.1.4 Hydrogen peroxide (H_2O_2).

3.1.5 Hydrofluoric acid (HF).

3.1.6 Strontium chloride (SrCl_2): excellent-grade of purity.

3.2 Reagent Preparation

3.2.1 Mixed acid: nitric acid + perchloric acid (4 + 1).

3.2.2 Strontium chloride solution (15 g/L): weigh-take 15 g of strontium chloride, then, add 100 mL of water and 45 mL of hydrochloric acid to dissolve it. Use water to dilute it to the constant volume of 1,000 mL.

3.3 Standards

Magnesium oxide (MgO, CAS No.: 1309-48-4): purity $\geq 99.9\%$, or magnesium element solution standard substance.

3.4 Preparation of Standard Solution

3.4.1 Magnesium Element Standard Stock Solution

Heat magnesium oxide at high temperature of 750 °C for 2 h, then, cool it down. Weigh-take 1.658 g of magnesium oxide, then, place it in a beaker. Add 50 mL of water and 45 mL of hydrochloric acid to dissolve it. Use water to dilute to the constant volume of 1,000 mL; mix it up. The concentration of magnesium element shall be 1,000 mg/L. Store the stock solution at 4 °C; it can be stored for 1 year.

3.4.2 Magnesium Element Standard Intermediate Solution

Accurately absorb magnesium element standard stock solution or magnesium element solution standard substance. Use strontium chloride solution to dilute it; prepare it into 100 mg/L magnesium element standard intermediate solution. Store the intermediate solution at 4 °C; it can be stored for 3 months.

3.4.3 Magnesium Element Standard Working Solution

Respectively and accurately absorb 0 mL, 0.5 mL, 1 mL, 2 mL, 4 mL and 5 mL of magnesium element standard intermediate solution, then, place it in a 100 mL volumetric flask. Use strontium chloride solution to dilute it to the constant volume; prepare it into magnesium element standard working solution at the concentration of 0 mg/L, 0.5 mg/L, 1 mg/L, 2 mg/L, 4 mg/L and 5 mg/L.

4 Instruments and Equipment

4.1 Flame atomic absorption spectrophotometer (equipped with magnesium hollow cathode lamp or electrodeless discharge lamp).

4.2 Balance: division value: 0.1 mg and 0.01 g.

4.3 Microwave digestion instrument (equipped with PTFE inner tank).

4.4 PTFE crucible: 250 mL, with a cover.

properly dilute it, then, determine it. In terms of sample whose talcum powder content is around 10 g/kg, dilute by 10 times.

5.2.2 Microwave digestion method

Weigh-take 0.5 g of sample (accurate to 0.01 g), place it into a digestion tank. Add 7 mL of nitric acid and 1 mL of hydrogen peroxide, soak it overnight, or conduct pre-digestion. Use microwave digestion instrument for microwave digestion. Please refer to Appendix A for reference conditions of microwave digestion. After cooling it down, open the digestion tank. Use 20 mL of water to rinse the digestion tank. Completely transfer the liquid and solid residue in the tank onto quantitative filter paper. Filter it, then, use 100 mL of water to rinse the filter paper and filter residue. Place the filter paper and residue into the digestion tank, then, heat it up to remove water content in the filter paper. Add 7 mL of nitric acid, 1 mL of hydrogen peroxide and 3 mL of hydrofluoric acid into the digestion tank. Use microwave digestion instrument for microwave digestion. After cooling it down, open the digestion tank. Heat it up to drive away acid. Wait till the fluid in the digestion tank is nearly dry, take the digestion tank down; cool it down. Use strontium chloride solution to rinse the digestion tank. Transfer the digestive juice into a 100 mL volumetric flask. Use strontium chloride solution to dilute to the constant volume; mix it up. Consider this solution as sample solution; reserve it for later usage. In terms of sample solution which has a high content of talcum powder, properly dilute it, then, determine it. In terms of sample whose talcum powder content is around 10 g/kg, dilute by 10 times.

5.3 Blank Test

Blank test shall be conducted simultaneously with sample digestion. Use the sample to be determined in the blank test. Conduct sample digestion in accordance with the same steps as sample digestion. Hydrofluoric acid shall not be added in the digestion of the filter paper and solid residue on the paper; talcum powder will not trigger chemical reaction in the sample. The sampling size, constant volume and the times of dilution in the blank test shall be the same as sample digestion.

5.4 Instrument Reference Conditions

- a) Wavelength: 285.2 nm;
- b) Spectral bandwidth: 0.5 nm;
- c) Lamp current: 4.0 mA;
- d) Air flow: 13.5 L/min;
- e) Acetylene flow: 2.0 L/min;
- f) Burner height: 7.0 mm.

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