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GB

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 29209-2012

National food safety standard - Food additive - Sodium sulfate

食品安全国家标准 食品添加剂 硫酸钠

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Table of Contents

Appendix A Testing method		5
3	Technical requirements	3
2	Chemical name, molecular formula, and relative molecular mass	3
1	Scope	3

National food safety standard - Food additive - Sodium sulfate

1 Scope

This standard applies to food additive sodium sulfate produced from raw materials such as mirabilite.

2 Chemical name, molecular formula, and relative molecular

mass

2.1 Chemical name

Anhydrous sodium sulfate

Sodium sulfate decahydrate

2.2 Molecular formula

Anhydrous sodium sulfate: Na₂SO₄

Sodium sulfate decahydrate: Na₂SO₄ • 10H₂O

2.3 Relative molecular mass

Anhydrous sodium sulfate: 142.04 (according to the 2007 International Relative Atomic

Mass)

Sodium sulfate decahydrate: 322.20 (according to the 2007 International Relative

Atomic Mass)

3 Technical requirements

3.1 Sensory requirements

Sensory requirements shall meet the requirements in Table 1.

Appendix A

Testing method

A.1 Warning

Some of the reagents used in the test methods of this standard are toxic or corrosive, and appropriate safety and protective measures shall be taken during operation.

A.2 General provisions

Unless otherwise specified, the reagents and water used in this standard refer to analytical reagents and third-grade water specified in GB/T 6682-2008. The standard solutions, preparations, and products used in the test for the determination of impurities shall be prepared in accordance with the provisions of GB/T 602 and GB/T 603 unless otherwise specified. The solution used refers to the aqueous solution when the solvent used for preparation is not indicated.

A.3 Identification test

A.3.1 Reagents and materials

- **A.3.1.1** Hydrochloric acid.
- **A.3.1.2** Potassium carbonate solution: 150 g/L.
- **A.3.1.3** Potassium pyroantimonate solution: Weigh 2 g of potassium pyroantimonate, dissolve it in 95 mL of hot water, and cool it rapidly; add 50 mL of potassium hydroxide solution (50 g/L) and 1 mL of sodium hydroxide solution (85 g/L), let it stand for 24 h, then filter it and add water to 150 mL.
- **A.3.1.4** Barium chloride (BaCl₂ 2H₂O) solution: 122 g/L.
- **A.3.1.5** Lead acetate solution: 95 g/L.
- **A.3.1.6** Platinum wire.

A.3.2 Identification method

A.3.2.1 Identification of sodium ions

A.3.2.1.1 Weigh about 0.1 g of the sample and dissolve it in 2 mL of water; add 2 mL of potassium carbonate solution, heat to boiling, and there shall be no precipitation. Add 4 mL of potassium pyroantimonate solution and heat to boiling. Cool it in ice water, rub

the inner wall of the test tube with a glass rod if necessary, and there shall be a white precipitate.

A.3.2.1.2 Take a platinum wire, moisten it with the hydrochloric acid solution, and burn it in a colorless flame until it is colorless; then, dip a little sample solution and burn it on a colorless flame, the flame shall become bright yellow.

A.3.2.2 Identification of sulfate ion

- **A.3.2.2.1** Take about 0.5 g of the sample, dissolve it in 10 mL of water, add a barium chloride solution, and there shall be a white precipitate, which is insoluble in hydrochloric acid solution.
- **A.3.2.2.** Take about 0.5 g of the sample, dissolve it in 10 mL of water, add a lead acetate solution, and there shall be a white precipitate, which is soluble in the ammonium acetate solution.
- **A.3.2.2.3** Take about 0.5 g of the sample, dissolve it in 10 mL of water, add hydrochloric acid, and there shall be no precipitation.

A.4 Determination of sodium sulfate (Na₂SO₄) content

A.4.1 Method summary

The sample is dissolved in water, a barium chloride is added under acidic conditions then to form a barium sulfate precipitate with sulfate ions in the test solution; after filtering, washing, drying, washing, burning, and weighing, the content can be calculated.

A.4.2 Reagents and materials

- **A.4.2.1** Hydrochloric acid solution: 1+1.
- **A.4.2.2** Barium chloride (BaCl₂ 2H₂O) solution: 122 g/L.
- **A.4.2.3** Silver nitrate solution: 20 g/L.
- **A.4.2.4** Slow quantitative filter paper.

A.4.3 Instruments and equipment

- **A.4.3.1** Electric constant-temperature drying oven: 105 °C±2 °C.
- **A.4.3.2** High-temperature furnace: The temperature can be controlled at 800 °C±25 °C.

A.4.4 Analysis steps

Weigh about 5 g of the sample after being dried at 105 °C±2 °C for 4 h, and the weight

A.5.1.3 Nitric acid.

A.5.1.4 Sodium hydroxide solution: 250 g/L.

A.5.1.5 Ammonium pyrrolidine dithiocarbamate (APDC) solution: 20 g/L. Filter it before use.

A.5.1.6 Lead (Pb) standard solution: 0.01 mg/mL.

A.5.1.7 Water: It shall meet the provisions of the second-grade water in GB/T 6682-2008.

A.5.2 Instruments and equipment

Atomic absorption spectrophotometer: It shall be equipped with a lead hollow cathode lamp.

A.5.3 Analysis steps

A.5.3.1 Cleaning of glass instruments

All instruments shall be soaked in a nitric acid solution (1+2) overnight, rinsed repeatedly with tap water, and finally rinsed with water.

A.5.3.2 Preparation of lead standard solution

Pipette 2.00 mL of lead standard solution, place it in a 150 mL beaker, and add 1 mL of hydrochloric acid. Heat it to boil for 5 min, cool it to room temperature, and dilute it with water to 100 mL. Use sodium hydroxide solution to adjust the pH of the solution to 1.0~1.5 (check with precision pH test paper). Transfer this solution to a 250 mL separatory funnel and dilute it with water to about 200 mL. Add 2 mL of ammonium pyrrolidine dithiocarbamate (APDC) solution and mix well. Extract twice with chloroform, add 20 mL each time, and collect the extract liquor (that is, the organic phase) in a 50 mL beaker; evaporate it to dryness on a steam bath (this operation must be performed in a fume hood), add 3 mL nitric acid to the residue, and continue to evaporate it to near dryness. Add 0.5 mL of nitric acid and 10 mL of water, and heat it until the volume of the solution is about 3 mL~5 mL. Transfer it to a 10 mL volumetric flask, dilute it to the mark with water, and shake well.

A.5.3.3 Preparation of sample solution

Weigh about 10 g of the sample, and the weight shall be accurate to 0.01 g; place it in a 150 mL beaker, and add 30 mL of water and 1 mL of hydrochloric acid. Cover it with a watch glass and heat to boiling for 5 min, then cool. Dilute it to 100 mL with water. Use sodium hydroxide solution to adjust the pH of the solution to 1.0~1.5 (check with precision pH test paper). The subsequent operations are the same as the steps in A.5.3.2

A.6.3.2 Preparation of standard solution

Pipette 6.00 mL of the selenium standard solution and place it in a 150 mL beaker. The subsequent operations are the same as the steps in A.6.3.1 "add 25 mL hydrochloric acid solution... Collect the extract liquid".

A.6.3.3 Preparation of the blank sample solution

Except that no sample is added to the blank sample solution, the types and amounts of reagents added are the same as those in the sample solution.

A.6.3.4 Determination

Use a 1 cm cuvette, at a wavelength of about 380 nm, with the blank sample solution as a reference, and measure the absorbance; the absorbance of the sample solution shall be not greater than the absorbance of the standard solution.

A.7 Determination of loss on drying

A.7.1 Instruments and equipment

Electric constant-temperature drying oven: The temperature can be controlled at $105 \, ^{\circ}\text{C} \pm 2 \, ^{\circ}\text{C}$.

A.7.2 Analysis steps

Weigh 1 g~2 g sample with a weighing bottle that has been dried at 105 °C±2 °C to a constant mass, and the weight shall be accurate to 0.0002 g; dry it in an electric constant-temperature drying oven at 105 °C±2 °C for 4 h.

A.7.3 Calculation of results

The mass fraction w_2 of loss on drying is calculated according to the formula (A.2):

$$w_2 = \frac{m - m_1}{m} \times 100\%$$
 (A. 2)

where:

m --- The value of the mass of the sample, in grams (g);

 m_1 --- The value of the mass of the sample after drying, in grams (g).

The experimental results shall be the arithmetic mean of the parallel measurement results. The absolute difference between the two independent measurement results obtained under repeatability conditions shall be not more than 0.2% for anhydrous sodium sulfate and not more than 1.0% for sodium sulfate decahydrate.

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