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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 25584-2010

**National food safety standards of food additives magnesium
chloride**

食品安全国家标准 食品添加剂 氯化镁

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National food safety standards of food additives magnesium chloride

1 Scope

This standard applies to magnesium chloride, a food additive made from bischofite or directly from salt production mother liquor.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) is applicable to this standard.

3 Molecular formula and relative molecular mass

3.1 Molecular formula

$\text{MgCl}_2 \cdot n\text{H}_2\text{O}$ ($n = 0, 6$)

3.2 Relative molecular mass

95.20 ($n = 0$) (according to the 2007 international relative atomic mass)

203.3 ($n = 6$) (according to the 2007 international relative atomic mass)

4 Technical requirements

4.1 Sensory requirements: It shall meet the requirements of Table 1.

Table 1 -- Sensory requirements

Item	Requirements	Inspection methods
Color	White	Take an appropriate amount of specimen; place it in a 50 mL beaker; observe the color under natural light
Organic state	Flake or granular crystals	

4.2 Physical and chemical indicators: It shall comply with the requirements of Table 2.

Appendix A

(Normative)

Test method

A.1 Warning

Some reagents used in the test method of this standard are corrosive, so the operator must be careful! If splashed on the skin, rinse with water immediately; seek medical attention immediately in severe cases. Operations using volatile organic solvents shall be carried out in a fume hood.

A.2 General provisions

The reagents and water used in the test method of this standard, unless otherwise specified, refer to analytical reagents and grade 3 water specified in GB/T 6682-2008. The standard titration solution, impurity standard solution, preparation and product required in the test, unless otherwise specified, shall be prepared in accordance with the provisions of HG/T 3696.1, HG/T 3696.2, HG/T 3696.3.

A.3 Identification test

A.3.1 Reagents and materials

A.3.1.1 Ammonia solution: 2 + 3.

A.3.1.2 Ammonium chloride solution: 100 g/L.

A.3.1.3 Ammonium carbonate solution: 100 g/L.

A.3.1.4 Silver nitrate solution: 42 g/L.

A.3.1.5 Disodium hydrogen phosphate solution ($\text{Na}_2\text{HPO}_3 \cdot 12\text{H}_2\text{O}$): 160 g/L.

A.3.2 Analytical procedures

A.3.2.1 Identification of magnesium ions

Take about 0.2 g of the specimen; dissolve it in 10 mL of water. No precipitation is produced when adding ammonium chloride solution and ammonium carbonate solution. When adding disodium hydrogen phosphate solution, a white precipitate shall be produced. Separate the precipitate and add ammonia solution. The precipitate shall not dissolve.

A.3.2.2 Identification of chlorides

Take about 0.2 g of the specimen; dissolve it in 10 mL of water. Add silver nitrate

solution to produce a white precipitate. This precipitate is insoluble in nitric acid but soluble in excess ammonia solution.

A.4 Determination of magnesium chloride

A.4.1 Method summary

Use triethanolamine to mask a small amount of trivalent iron, trivalent aluminum, divalent manganese ions. When the pH is 10, use chrome black T as an indicator and titrate the total amount of calcium and magnesium with disodium ethylenediaminetetraacetic acid standard titration solution. Subtract the calcium content from it, to calculate the magnesium chloride content.

A.4.2 Reagents and materials

A.4.2.1 Hydrochloric acid solution: 1 + 1.

A.4.2.2 Triethanolamine solution: 1 + 3.

A.4.2.3 Ammonia-ammonium chloride buffer solution (A) ($\text{pH} \approx 10$).

A.4.2.4 Silver nitrate solution: 10 g/L.

A.4.2.5 Disodium ethylenediaminetetraacetic acid standard titration solution: $c(\text{EDTA}) = 0.02 \text{ mol/L}$.

A.4.2.6 Chrome black T indicator.

A.4.3 Analytical procedure

A.4.3.1 Preparation of test solution A

Weigh about 10 g of the specimen, accurate to 0.000 2 g; place in a 250 mL beaker; dissolve in 20 mL of water. Add 1 mL of hydrochloric acid solution; heat to boiling; keep slight boiling for 1 min ~ 2 min. After cooling, transfer to a 250 mL volumetric flask; dilute to the mark with water; shake well. This solution is test solution A. This solution is retained for the determination of magnesium chloride content and calcium content.

A.4.3.2 Determination

Pipette 25.00 mL of test solution A; place in a 250 mL volumetric flask; dilute to the mark with water; shake well. Transfer 25.00 mL of the above solution into a 250 mL conical flask; add 50 mL of water, 5 mL of triethanolamine solution, 10 mL of ammonia-ammonium chloride buffer solution A, 0.1 g of chrome black T indicator; titrate with disodium ethylenediaminetetraacetic acid standard titration solution, until the solution changes from purple-red to pure blue.

c - The accurate value of the concentration of the standard titration solution of disodium ethylenediaminetetraacetic acid, in moles per liter (mol/L);

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

M₂ - The value of the molar mass of magnesium chloride (MgCl₂), in grams per mole (g/mol) (M₂ = 95.20).

The arithmetic mean of the parallel determination results is taken as the determination result; the absolute difference between the two parallel determination results is not greater than 0.1%.

A.5 Determination of calcium

A.5.1 Reagents and materials

A.5.1.1 Sodium hydroxide solution: 100 g/L.

A.5.1.2 Triethanolamine solution: 1 + 3.

A.5.1.3 Disodium ethylenediaminetetraacetic acid standard titration solution: c (EDTA) = 0.02 mol/L.

A.5.1.4 Calcium reagent sodium carboxylate indicator.

A.5.2 Instruments and equipment

Micro-burette: Graduation value is 0.02 mL.

A.5.3 Analytical procedure

Pipette 50.00 mL of test solution A into a 250 mL conical flask; add 30 mL of water and 5 mL of triethanolamine solution; add sodium hydroxide solution dropwise while shaking. When the solution just begins to precipitate, add 0.1 g of calcium reagent sodium carboxylate indicator; continue to add sodium hydroxide solution dropwise until the solution changes from blue to wine red, with an excess of 0.5 mL. Titrate with disodium ethylenediaminetetraacetic acid standard titration solution, until the solution changes from wine red to pure blue.

A.5.4 Calculation of results

Calcium content is calculated as the mass fraction w_3 of calcium (Ca), which is expressed in % and calculated according to formula (A.3):

$$w_3 = \frac{(V_2/1000)cM}{m \times 50/500} \times 100 \dots\dots\dots (A.3)$$

Where:

V_2 - The volume of disodium ethylenediaminetetraacetic acid standard titration solution consumed by titrating the test solution, in milliliters (mL);

c - The exact concentration of disodium ethylenediaminetetraacetic acid standard titration solution, in moles per liter (mol/L);

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

M - The molar mass of calcium (Ca), in grams per mole (g/mol) ($M = 40.01$).

The arithmetic mean of the parallel determination results is taken as the determination result; the absolute difference between the two parallel determination results is not greater than 0.02%.

A.6 Determination of sulfate

A.6.1 Reagents and materials

A.6.1.1 Hydrochloric acid solution: 1 + 1.

A.6.1.2 Nitric acid solution: 1 + 4.

A.6.1.3 Barium chloride solution: 100 g/L.

A.6.1.4 Silver nitrate solution: 20 g/L.

A.6.1.5 Methyl red indicator solution: 2 g/L.

A.6.2 Instruments and equipment

High temperature furnace: It can control the temperature of $850\text{ }^{\circ}\text{C} \pm 50\text{ }^{\circ}\text{C}$.

A.6.3 Analytical procedures

A.6.3.1 Preparation of test solution B

Weigh about 25 g of specimen, accurate to 0.01 g; add appropriate amount of water to dissolve; add 2 mL of hydrochloric acid solution; place on an electric furnace and heat to boiling; keep it boiling for 1 min ~ 2 min. After cooling, pipette to a 250 mL volumetric flask; dilute with water to the scale; shake well. Dry filter with medium-speed qualitative filter paper and discard 20 mL of the initial filtrate. The filtrate is used as test solution B.

A.6.3.2 Determination

Pipette 25.00 mL of test solution B into a 250 mL beaker; add water to 50 mL; add 1 mL of hydrochloric acid solution; heat to boil on an electric furnace; add 5 mL of barium chloride solution dropwise while stirring for about 1 minute. Continue stirring and

leave for 40 min to eliminate bubbles. Place the specimen solution colorimetric tube and the standard solution colorimetric tube on a white background; compare visually along the axis of the colorimetric tube. The chromaticity produced by the specimen solution shall not be greater than the chromaticity produced by the standard solution.

A.9 Determination of lead

A.9.1 Reagents and materials

A.9.1.1 Chloroform.

A.9.1.2 Nitric acid.

A.9.1.3 Hydrochloric acid solution: 1 + 4.

A.9.1.4 Sodium hydroxide solution: 100 g/L.

A.9.1.5 Ammonium pyrrolidine dithiocarbamate (APDC) solution: 20 g/L.

Weigh $2.00\text{ g} \pm 0.01\text{ g}$ of ammonium pyrrolidine dithiocarbamate (APDC); dissolve it in 100 mL of water. If there is insoluble matter, filter it before use.

A.9.1.6 Lead standard solution: 1 mL of solution contains 0.010 mg of lead (Pb).

Use a pipette to transfer 1.00 mL of the lead standard solution, which is prepared according to HG/T 3696.2; place it in a 100 mL volumetric flask; add water to the mark; shake well. The solution is prepared immediately before use.

A.9.1.7 Precision pH test paper: 0.5 ~ 5.0.

A.9.2 Instruments and equipment

A.9.2.1 Separatory funnel: 250 mL.

A.9.2.2 Atomic absorption spectrophotometer: Equipped with a lead hollow cathode lamp.

A.9.3 Determination steps

A.9.3.1 Preparation of lead standard determination solution

Pipette 1.00 mL of the lead standard solution; place it in a 150 mL beaker. Use hydrochloric acid solution to adjust the pH of the solution to 1.0 ~ 1.5 (check with precision pH test paper). Transfer the solution into a separatory funnel; dilute it with water to about 200 mL. Add 2 mL of ammonium pyrrolidine dithiocarbamate (APDC) solution; shake well. Extract twice with chloroform, adding 20 mL each time; collect the extract (i.e., organic phase) in a 50 mL beaker; evaporate to dryness in a water bath in a fume hood. Add 3 mL of nitric acid to the residue; heat to near dryness. Add 0.5

mL of nitric acid and 10 mL of water; heat until the remaining liquid volume is 3 mL ~ 5 mL; transfer to a 10 mL volumetric flask; dilute to the mark with water.

A.9.3.2 Preparation of test solution

Weigh $10.00\text{ g} \pm 0.01\text{ g}$ of the specimen; place it in a 150 mL beaker; add 30 mL of water; add 10 mL of hydrochloric acid solution; cover with a watch glass and heat to boiling for 5 min. Cool and adjust the pH value to 1.0 ~ 1.5 with sodium hydroxide solution (check with precision pH test paper). Then proceed as in A.9.3.1, from "Transfer the solution into the separatory funnel..."

A.9.3.3 Determination

Use air-acetylene flame, to adjust to zero with water at a wavelength of 283.3 nm; use an atomic absorption spectrophotometer to determine the absorbance of the lead standard solution and the test solution.

A.9.4 Result determination

The absorbance of the test solution shall not be greater than the absorbance of the lead standard solution.

A.10 Determination of arsenic

Weigh $2.00\text{ g} \pm 0.01\text{ g}$ of the specimen; place it in a 250 mL beaker; add 50 mL of water; add 10 mL of hydrochloric acid as the test solution.

Preparation of limit standard solution: Pipette 1.00 mL of arsenic standard solution [1 mL of solution contains arsenic (As) 0.001 mg]; perform the following determination according to Chapter 11 of GB/T 5009.76-2003.

A.11 Determination of ammonium

A.11.1 Reagents and materials

A.11.1.1 Nessler's reagent.

A.11.1.2 Sodium hydroxide solution: 100 g/L.

A.11.1.3 Potassium sodium tartrate ($\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$) solution: 500 g/L.

A.11.1.4 Ammonium standard solution: 1.00 mL of solution contains 0.02 mg of ammonium (NH_4).

Use a pipette to take 2.00 mL of the ammonium standard solution prepared according to HG/T 3696.2; dilute it to 100 mL with water; shake well. The solution is prepared before use.

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