GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 5009.42-2016

National Food Safety Standard -

Determination of Table Salt Index

食品安全国家标准

食盐指标的测定

Issued on: August 31, 2016 **Implemented on: March 01, 2017**

Issued by: National Health and Family Planning Commission of the PRC

Table of Contents

National Food Safety Standard - Determination of Table Salt Index

1 Scope

This Standard specifies the testing methods of sodium chloride, lead, total arsenic, cadmium, total mercury, barium, potassium chloride, potassium ferrocyanide, and iodine in table salt.

This Standard applies to the determination of table salt index.

2 Determination of sodium chloride

2.1 Determination of water content

Operate according to the direct drying method in GB 5009.3. The drying temperature is set to 140 °C±2 °C. DRY until the difference between the two masses before and after does not exceed 5 mg, that is, constant weight.

2.2 Determination of chloride ion

2.2.1 Principle

After the sample is dissolved, USE potassium chromate as an indicator; USE a silver nitrate standard titration solution to titrate, to determine the content of chloride ion.

2.2.2 Reagents and materials

Unless otherwise stated, the reagents used in this method are analytically pure; the water is the Grade 3 water specified in GB/T 6682.

2.2.2.1 Reagents

2.2.2.1.1 Silver nitrate.

2.2.2.1.2 Potassium chromate.

2.2.2.2 Preparation of reagents

2.2.2.2.1 Silver nitrate standard titration solution (0.1 mol/L).

liter (mol/L);

35.453 - The molar mass of chloride ion, in grams per mole (g/mol);

f - The dilution factor of the sample solution;

m - Sample mass, in grams (g);

100, 1000 - Unit conversion factor.

The calculation result is retained to two decimal places.

2.2.6 Precision

The absolute difference between the two independent determination results, obtained under repeated conditions, shall not exceed 5% of the arithmetic mean.

2.3 Determination of calcium

Operate according to the atomic absorption spectrophotometry in GB/T 5009.92.

2.4 Determination of magnesium

Operate according to GB/T 5009.90.

2.5 Determination of sulfate radical (EDTA complexometric titration)

2.5.1 Principle

Excess barium chloride and the sulfate radical in the sample form insoluble barium sulfate precipitate. The remaining barium ions are titrated using a standard solution of disodium edetate (EDTA). USE indirect method to determine the sulfate radical.

2.5.2 Reagents and materials

Note: Unless otherwise stated, the reagents used in this method are analytically pure; the water is the Grade 3 water specified in GB/T 6682.

2.5.2.1 Reagents

2.5.2.1.1 Hydrochloric acid.

2.5.2.1.2 Aqueous ammonia.

2.5.2.1.3 Ammonium chloride.

2.5.2.1.4 Absolute ethanol.

2.5.4 Analytical procedures

2.5.4.1 Sample processing

WEIGH 25 g (accurate to 0.001 g) of the pulverized sample in a 400 mL beaker; ADD about 200 mL of water; HEAT on a boiling water bath; USE a glass rod to stir until completely dissolved. After cooling, TRANSFER to a 500 mL volumetric flask; ADD water to the mark; SHAKE well; if necessary, filter. When the content of ions to be tested in the sample is too high, it may be appropriately diluted and then determined.

2.5.4.2 Determination

PIPETTE a sample solution (2.5.4.1) of a certain volume (so that the sulfate radical content in the solution is below 8 mg) in a 150 mL conical flask. ADD 1 drop of hydrochloric acid solution (2.5.2.2.1); ADD 5.00 mL of barium chloride solution (2.5.2.2.6); STIR for a while; LET it stand for 5 min. ADD 5 mL of ethylenediaminetetraacetic acid disodium magnesium solution, 10 mL of absolute ethanol, 5 mL of ammonia-ammonium chloride buffer solution, 4 drops of chrome black T indicator. USE EDTA standard titration solution to titrate, until the solution changes from wine red to brilliant blue. RECORD the volume V_3 of EDTA standard titration solution consumed.

Titration of the total amount of calcium-magnesium in the solution: PIPETTE the sample solution of the same volume as the sulfate radical to be determined; PLACE it in a 150 mL conical flask. ADD water to 25 mL. ADD 5 mL of ammonia-ammonium chloride buffer solution, 4 drops of chrome black T indicator. USE EDTA standard titration solution to titrate, until the solution changes from wine red to brilliant blue. RECORD the volume V_2 of EDTA standard solution consumed.

2.5.5 Expression of analysis results

The content of sulfate radical in the sample is calculated according to formula (2):

$$X_2 = \frac{(V_1 + V_2 - V_3) \times c \times 96.06 \times F}{m \times 1\,000} \times 100 \quad \cdots\cdots (2)$$

Where:

X_2 - The content of sulfate radical in the sample, %;

V_1 - The amount of EDTA standard titration solution used when titrating the barium chloride solution, in milliliters (mL);

2.6.2 Expression of analysis results

2.6.2.1 Sodium chloride (wet basis)

The content $X_{(w)}$ of sodium chloride (wet basis) in the sample is the sodium chloride content calculated in accordance with 2.6.1, in %.

The calculation result is retained to two decimal places.

2.6.2.2 Sodium chloride (on a dry basis)

The sodium chloride content in the sample is calculated according to formula (3):

$$X_3 = \frac{X_{(w)}}{1 - P} \qquad \cdots\cdots (3)$$

Where:

X_3 - The content of sodium chloride (on a dry basis) in the sample, %;

$X_{(w)}$ - The content of sodium chloride (wet basis) in the sample, %;

P - Water content, %.

The calculation result is retained to two decimal places.

2.6.3 Precision

The absolute difference between the two independent determination results, obtained under repeated conditions, shall not exceed 10% of the arithmetic mean.

3 Determination of lead

3.1 Principle

After the sample is treated, the lead ions, at a certain pH, form a complex with sodium diethyldithiocarbamate (DDTC). It is extracted and separated by 4-methyl-2-pentanone, introduced into an atomic absorption spectrometer. After electrothermal atomization, absorb the 283.3 nm resonance line. In a certain concentration range, the absorption value is directly proportional to the lead content, in comparative quantification with the standard series.

3.2 Reagents and materials

in a 100 mL volumetric flask; USE nitric acid solution (3.2.2.7) to dilute to the mark. In such a way, it is diluted several times to a standard use solution containing 0.0 ng, 5.0 ng, 10.0 ng, 20.0 ng, and 40.0 ng of lead per milliliter, respectively.

3.3 Instruments and equipment

3.3.1 Atomic absorption spectrometer, with graphite furnace and lead hollow cathode lamp.

3.3.2 Balance: The sensitivity is 0.001 g.

3.3.3 Adjustable electric hot plate and adjustable electric furnace.

3.4 Analytical procedures

3.4.1 Sample processing

Accurately WEIGH 10 g (accurate to 0.01 g) of sample in a 100 mL beaker; ADD a small amount of water to dissolve; ADD a small amount of mixed acid (3.2.2.1); HEAT to boil; LET cool and TRANSFER all to a 50 mL volumetric flask; DILUTE to the mark; MIX well for use.

3.4.2 Extraction separation

Depending on the sample, PIPETTE 25.0 mL~50.0 mL of the sample solution prepared according to 3.4.1 and the reagent blank solution into 125 mL separating funnels respectively; ADD water to 60 mL. ADD 2 mL of ammonium citrate solution (3.2.2.3), 3 drops~5 drops of bromothymol blue solution (3.2.2.4). USE aqueous ammonia solution (3.2.2.6) to adjust the pH, until the solution turns from yellow to blue. ADD 10.0 mL of ammonium sulfate solution (3.2.2.2), 10 mL of DDTC solution (3.2.2.5); SHAKE well. PLACE it for about 5 min; ADD 10.0 mL of MIBK (3.2.1.8); SHAKE vigorously to extract for 1 min. After letting stand for stratification, DISCARD the aqueous layer; PUT the MIBK layer into a 10 mL stoppered graduated tube for use. PIPETTE 10.0 mL of lead standard use solution into the 125 mL separating funnels respectively. EXTRACT in the same manner as the sample. At the same time, DO reagent blank.

3.4.3 Determination

It is same as the determination of graphite furnace atomic absorption spectrometry in GB 5009.12.

3.5 Expression of analysis results, precision

It is same as the expression of analysis results and precision of graphite furnace atomic absorption spectrometry in GB 5009.12.

7.3 Instruments and equipment

Balance: The sensitivity is 0.001 g.

7.4 Analytical procedures

WEIGH 50.00 g of the sample; ADD water to dissolve to 500 mL; filter. DISCARD the primary filtrate; MEASURE 50 mL of the filtrate in a 50 mL colorimetric tube. TAKE 1 mL of barium standard use solution (7.2.2.3) in a 50 mL colorimetric tube; ADD water to the mark; MIX well. ADD 2 mL of sulfuric acid solution (7.2.2.1) to each of the two tubes; SHAKE well. After placing for 2 h, visually compare them. The sample tube shall not be turbid than the standard tube, that is, ≤15 mg/kg barium.

8 Determination of potassium chloride

Note: When the potassium chloride content in the table salt is >2 g/100 g, operate according to the gravimetric method. When the potassium chloride content in the table salt is <2 g/100 g, operate according to flame emission spectroscopy.

8.1 Flame emission spectroscopy

8.1.1 Operate according to GB/T 5009.91.

8.1.2 Expression of analysis results

The content of potassium chloride in the sample is calculated according to formula (4):

$$X_4 = \frac{(c - c_0) \times V \times f \times 1.906\ 6}{m \times (1 - P) \times 1\ 000 \times 1\ 000} \times 100 \quad \cdots\cdots (4)$$

Where:

X_4 - The content of potassium chloride (on a dry basis) in the sample, in grams per hundred grams (g/100 g);

c - The concentration of potassium in the sample solution for determination (obtained from the standard curve), in micrograms per milliliter (μg/mL);

c_0 - The concentration of potassium in the reagent blank solution (obtained from the standard curve), in micrograms per milliliter (μg/mL);

V - The constant volume of the sample solution, in milliliters (mL);

f - The dilution factor of the sample solution;

it. ADD 5 g of aluminum hydroxide; STIR for 10 min; USE slow filter paper to filter. If the filtrate is turbid, it shall be repeatedly filtered to clarification. COLLECT all the filtrate into a 250 mL volumetric flask; ADD 1 mL of sodium hydroxide solution; DILUTE to the mark; SHAKE well; re-filter before use.

8.2.2.2.4 Sodium tetraphenylborate washing solution (1 g/L): PIPETTE 20 mL of sodium tetraphenylborate solution into a 500 mL volumetric flask; DILUTE to the mark; SHAKE well.

8.2.2.2.5 Phenolphthalein indicator solution (5 g/L): WEIGH 0.5 g of phenolphthalein dissolved in 100 mL of 95% ethanol solution.

8.2.3 Instruments and equipment

8.2.3.1 No. 4 glass sand core funnel (The aperture of filter plate is 5 μm~15 μm).

8.2.3.2 Circulating-water vacuum pump.

8.2.3.3 Electrothermal constant-temperature drying oven.

8.2.3.4 Dryer: Include an effective desiccant.

8.2.3.5 Analytical balance: The sensitivity is 0.1 mg.

8.2.4 Analytical procedures

WEIGH 2.5 g (accurate to 0.0001 g) of sample; PLACE it in a 100 mL beaker; ADD water to dissolve it; TRANSFER to a 500 mL volumetric flask; USE water to dilute to the mark; SHAKE well and filter; DISCARD the primary filtrate.

Accurately PIPETTE 25.0 mL of filtrate (The potassium chloride content in the sample solution shall not exceed 48 mg) in a 100 mL beaker. ADD 10 mL of EDTA solution, 2 drops of phenolphthalein indicator solution. With constant stirring, ADD dropwise the sodium hydroxide solution, until the color of the test solution turns pink, an excess of 1 mL; SHAKE well (At this time, the volume of the test solution is about 40 mL).

Under continuous stirring, ADD dropwise sodium tetraphenylborate solution of the volume 4 mL more than the theoretical amount (16 mg of potassium chloride requires 3 mL of sodium tetraphenylborate solution); LET it stand for 0.5 h.

USE a No. 4 glass sand core funnel which is pre-baked at 120 °C to a constant weight to suction-filter the precipitate. All the precipitate is washed using a sodium tetraphenylborate washing solution into the sand core funnel. USE the washing solution again to wash 5 times, 5 mL each time. Finally, USE water to wash twice, 2 mL each time. The sand core funnel and the precipitate are

9 Determination of potassium ferrocyanide (ferrous sulfate method)

9.1 Principle

Potassium ferrocyanide, under acidic conditions, forms a blue double salt with ferrous sulfate. It is in comparative quantification with the standard. The detection limit of the method is 1.0 mg/kg.

9.2 Reagents

Note: Unless otherwise stated, the reagents used in this method are analytically pure; the water is the Grade 3 water specified in GB/T 6682.

9.2.1 Reagents

9.2.1.1 Sulfuric acid.

9.2.1.2 Ferrous sulfate ($FeSO_4 \cdot 7H_2O$).

9.2.1.3 Potassium ferrocyanide.

9.2.2 Preparation of reagents

9.2.2.1 Sulfuric acid solution: MEASURE 5.7 mL of sulfuric acid; POUR it into 50 mL of water. After cooling, ADD water to 100 mL.

9.2.2.2 Ferrous sulfate solution (80 g/L): WEIGH 8 g of ferrous sulfate dissolved in 100 mL of sulfuric acid solution; filter; STORE it in a brown reagent bottle at low temperature.

9.2.3 Preparation of standard solutions

9.2.3.1 Standard solution of potassium ferrocyanide: Accurately WEIGH 0.1993 g of potassium ferrocyanide ($K_4[Fe(CN)_6] \cdot 3H_2O$) dissolved in a small amount of water. TRANSFER to a 100 mL volumetric flask; ADD water to dilute to the mark. 1 mL of this solution is equivalent to 1.0 mg of ferrocyanide ($[Fe(CN)_6]^{4-}$).

9.2.3.2 Standard working solution of potassium ferrocyanide: PIPETTE 10.0 mL of standard solution of potassium ferrocyanide; PUT it in a 100 mL volumetric flask; ADD water to dilute to the mark. 1 mL of this solution is equivalent to 0.10 mg of ferrocyanide ($[Fe(CN)_6]^{4-}$).

9.3 Instruments and equipment

9.6 Precision

The absolute difference between the two independent determination results, obtained under repeated conditions, shall not exceed 10% of the arithmetic mean.

10 Determination of iodine (redox titration method)

10.1 Principle

The iodide ion in the sample, under acidic conditions, is oxidized to iodate using sodium hypochlorite. Oxalic acid removes excess sodium hypochlorite. Iodate oxidizes potassium iodide to free elemental iodine. USE the starch solution as an indicator. USE a standard solution of sodium thiosulfate to titrate. Calculate the iodine content.

10.2 Reagents

Note: Unless otherwise stated, the reagents used in this method are analytically pure; the water is the Grade 3 water specified in GB/T 6682.

10.2.1 Reagents

10.2.1.1 Oxalic acid.

10.2.1.2 Phosphoric acid (ρ=85%).

10.2.1.3 Potassium iodide.

10.2.1.4 Sodium hypochlorite reagent solution (10% of available chlorine).

10.2.1.5 Starch.

10.2.1.6 Sodium thiosulfate.

10.2.2 Preparation of reagents

10.2.2.1 Oxalic acid-phosphoric acid mixed solution: WEIGH 15 g of oxalic acid; ADD water to dissolve; ADD 34 mL of phosphoric acid; USE water to dilute to 500 mL.

10.2.2.2 Potassium iodide solution (50 g/L): WEIGH 25.0 g of potassium iodide; USE water to dissolve and dilute to 500 mL; STORE in a brown bottle. Prepare when used.

10.2.2.3 Sodium hypochlorite solution (about 3% of available chlorine): MEASURE 10 mL of sodium hypochlorite reagent solution; ADD 30 mL of water;

This is an excerpt of the PDF (Some pages are marked off intentionally)

------ The End ------