GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 5009.92-2016

National food safety standard - Determination of calcium in foods

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China Food and Drug Administration.

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Foreword

This Standard replaces the determination method for calcium in GB/T 5009.92-2003 “Determination of calcium in foods”, GB 5413.21-2010 “National food safety standard - Determination of calcium, iron, zinc, sodium, potassium, magnesium, copper and manganese in foods for infants and young children, milk and milk products”, GB/T 23375-2009 “Determination of copper , iron , zinc , calcium , magnesium and phosphorus content in vegetables and derived products”, GB/T 14609-2008 “Inspection of grain and oils - Determination of copper, iron, manganese, zinc, calcium, magnesium in cereals and derived products by atomic absorption and flame spectrophotometry”, GB/T 14610-2008 “Inspection of grain and oils - Determination of calcium in cereals and cereal products”, GB/T 9695.13-2009 “Meat and meat products - Method for Determination of calcium content” and NY 82.19-1988 “Determination of fruit juice - Determination of calcium and magnesium”.

Compare this Standard with GB/T 5009.92-2003, the main changes are as follows:

- MODIFY the standard name TO "National food safety standard - Determination of calcium in foods";

- ADD the microwave digestion, pressure tank digestion;

- MODIFY the flame atomic absorption spectrometry and EDTA titration;

- ADD the inductively coupled plasma emission spectrometry;

- ADD the inductively coupled plasma mass spectrometry.

National food safety standard - Determination of calcium in foods

1 Scope

This Standard specifies the determination of calcium in foods by flame atomic absorption spectrometry, titration, inductively coupled plasma atomic emission spectrometry and inductively coupled plasma mass spectrometry.

This Standard applies to the determination of calcium content in foods.

Method I Flame atomic absorption spectrometry

2 Principle

After the sample is digested, add lanthanum solution as the releasing agent, atomize by atomic absorption flame, and measure the absorbance value at 422.7 nm, which is proportional to the calcium content in a certain concentration range. Compare with the standard series.

3 Reagents and materials

Unless otherwise specified, the reagents used in this method are excellent regents, and the water is Grade 2 water as 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 Hydrochloric acid (HCl).

3.1.4 Lanthanum oxide (La_2O_3).

3.2 Preparation of reagents

3.2.1 Nitric acid solution (5 + 95): MEASURE 50 mL of nitric acid; ADD 950 mL of water; MIX well.

3.2.2 Nitric acid solution (1 + 1): MEASURE 500 mL of nitric acid; MIX well with 500

mL of water.

3.2.3 Hydrochloric acid solution (1 + 1): MEASURE 500 mL of hydrochloric acid; MIX well with 500 mL of water.

3.2.4 Lanthanum solution (20 g/L): WEIGH 23.45 g of lanthana; firstly, MOISTEN with a small amount of water and then ADD 75 mL of hydrochloric acid solution (1 + 1) to dissolve; TRANSFER to a 1000 mL volumetric flask; ADD water to the mark; MIX well.

3.3 Standard

Calcium carbonate ($CaCO_3$, CAS No. 471-34-1): Purity > 99.99 %, or calcium standard solution at a certain concentration certified by the state and awarded the standard substance certificate.

3.4 Preparation of standard solutions

3.4.1 Calcium standard stock solution (1000 mg/L): accurately WEIGH 2.4963 g (to the nearest 0.0001 g) of calcium carbonate; ADD hydrochloric acid solution (1 + 1) to dissolve; TRANSFER to a 1000 mL volumetric flask; ADD water to the mark; MIX well.

3.4.2 Calcium standard intermediate solution (100 mg/L): accurately PIPETTE 10 mL of calcium standard stock solution (1000 mg/L) into a 100 mL volumetric flask; ADD nitric acid solution (5 + 95) to the mark; MIX well.

3.4.3 Calcium standard series solutions: respectively PIPETTE 0 mL, 0.500 mL, 1.00 mL, 2.00 mL, 4.00 mL and 6.00 mL of calcium standard intermediate solution (100 mg/L) into 100 mL volumetric flasks; ADD 5 mL of lanthanum solution (20 g/L) to each volumetric flask; and finally ADD nitric acid solution (5 + 95) to the mark; MIX well. The concentrations of calcium in standard calcium series solutions are 0 mg/L, 0.500 mg/L, 1.00 mg/L, 2.00 mg/L, 4.00 mg/L and 6.00 mg/L, respectively.

NOTE: It may determine the specific concentration of elements in the standard solution series according to the sensitivity of the instrument and the actual content of calcium in the sample.

4 Instruments and equipment

NOTE: All glassware and polytetrafluoroethylene digestion inner tank must be soaked in nitric acid solution (1 + 5) overnight, washed repeatedly with tap water, and finally rinsed with water.

4.1 Atomic absorption spectrometer: with flame atomizer, calcium hollow cathode lamp.

4.2 Analytical balance: with the division of 1 mg and 0.1 mg.

4.3 Microwave digestion system: with polytetrafluoroethylene digestion inner tank.

4.4 Adjustable electric furnace.

5.2.2 Microwave digestion

Accurately WEIGH 0.2 g ~ 3 g (to the nearest 0.001 g) of solid sample or accurately PIPETTE 0.500 mL ~ 3.00 mL of liquid sample in a microwave digestion tank; ADD 5 mL of nitric acid. DIGEST the sample according to procedure of the microwave digestion, refer to Annex A for digestion conditions. REMOVE the digestion tank after cooling; PLACE it on the electric hot plate to get rid of acid to about 1 mL at 140 °C ~ 160 °C. After the digestion tank is cool, TRANSFER the digestion solution to a 25 mL volumetric flask; WASH the digestion tank twice or three times with a small amount of water; COLLECT the washing liquid in the volumetric flask and ADD water to the mark. DILUTE according to the actual measurement needs; and ADD a certain volume of lanthanum solution (20 g/L) in the diluted solution, so that its concentration in the final diluted solution is 1 g/L; MIX for use, this is the sample solution to be tested. At the same time, carry out the reagent blank test.

5.2.3 Pressure tank digestion

Accurately WEIGH 0.2 g ~ 1 g (to the nearest 0.001 g) of solid sample or accurately PIPETTE 0.500 mL ~ 5.00 mL of liquid sample in a digestion inner tank; ADD 5 mL of nitric acid. COVER the inner cap; TIGHTEN the stainless-steel outer tank; PLACE into the constant temperature oven; MAINTAIN at 140 °C ~ 160 °C for 4 h ~ 5 h. Slowly LOOSEN the outer tank after cooling; REMOVE the digestion inner tank; PLACE it on the adjustable electric hot plate to get rid of acid to about 1 mL at 140 °C ~ 160 °C. After cooling, TRANSFER the digestion solution to a 25 mL volumetric flask; WASH the inner tank and the inner cap twice or three times with a small amount of water; COLLECT the washing liquid in the volumetric flask and ADD water to the mark; MIX well for use. DILUTE according to the actual measurement needs; and ADD a certain volume of lanthanum solution (20 g/L) in the diluted solution, so that its concentration in the final diluted solution is 1 g/L; MIX for use, this is the sample solution to be tested. At the same time, carry out the reagent blank test.

5.2.4 Dry ashing

Accurately WEIGH 0.5 g ~ 5 g (to the nearest 0.001 g) of solid sample or accurately PIPETTE 0.500 mL ~ 10.0 mL of liquid sample in a crucible; HEAT with low flame; CARBONIZE to smokeless. TRANSFER to a muffle furnace; ASH at 550 °C for 3 h ~ 4 h. COOL and REMOVE. For incompletely-ashed sample, ADD a few drops of nitric acid; HEAT with low flame; carefully EVAPORATE to dryness; TRANSFER to a muffle furnace at 550 °C; continue to ASH for 1 h ~ 2 h until the sample is grayish white; COOL and REMOVE; DISSOLVE with an appropriate amount of nitric acid solution (1 + 1) and TRANSFER to a graduated tube; ADD water to 25 mL. DILUTE according to the actual measurement needs; and ADD a certain volume of lanthanum solution in the diluted solution, so that its concentration in the final diluted solution is 1 g/L; MIX for use, this is the sample solution to be tested. At the same time, carry out the reagent blank test.

10.2.1 Potassium hydroxide solution (1.25 mol/L): WEIGH 70.13 g of potassium hydroxide; DILUTE with water to 1000 mL; MIX well.

10.2.2 Sodium sulfide solution (10 g/L): WEIGH 1 g of sodium sulfide; DILUTE with water to 100 mL; MIX well.

10.2.3 Sodium citrate solution (0.05 mol/L): WEIGH 14.7 g of sodium citrate; DILUTE with water to 1000 mL; MIX well.

10.2.4 EDTA solution: WEIGH 4.5 g of EDTA; DILUTE with water to 1000 mL; MIX well. STORE in polyethylene bottles at 4 °C. DILUTE 10 times when use.

10.2.5 Calcium red indicator: WEIGH 0.1 g of calcium red indicator; DILUTE with water to 100 mL; MIX well.

10.2.6 Hydrochloric acid solution (1 + 1): MEASURE 500 mL of hydrochloric acid; MIX well with 500 mL of water.

10.3 Standard

Calcium carbonate ($CaCO_3$, CAS No. 471-34-1): with purity > 99.99 %, or calcium standard solution at a certain concentration certified by the state and awarded the standard substance certificate.

10.4 Preparation of standard solution

Calcium standard stock solution (100.0 mg/L): accurately WEIGH 0.2496 g (to the nearest 0.0001 g) of calcium carbonate; ADD hydrochloric acid solution (1 + 1) to dissolve; TRANSFER to a 1000 mL volumetric flask; ADD water to the mark; MIX well.

11 Instruments and equipment

NOTE: All glassware and polytetrafluoroethylene digestion inner tank must be soaked in nitric acid solution (1 + 5) overnight, washed repeatedly with tap water, and finally rinsed with water.

11.1 Analytical Balance: with the division of 1 mg and 0.1 mg.

11.2 Adjustable electric furnace.

11.3 Adjustable electric hot plate.

11.4 Muffle furnace.

12 Analysis procedure

12.1 Sample preparation

V_1 - the volume of 10-fold diluted EDTA solution that is consumed when titrating the sample solution, in milliliters (mL);

V_0 - the volume of 10-fold diluted EDTA solution that is consumed when titrating the blank solution, in milliliters (mL);

V_2 - the constant volume of the sample digestion solution, in milliliters (mL);

1 000 - the conversion factor;

m - the mass of the sample weighed or the volume pipetted, in grams or milliliters (g or mL);

V_3 - the volume of the sample solution to be tested for titration, in milliliters (mL).

The calculation results retain three significant figures.

14 Precision

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

15 Others

When weighing 4 g (or 4 mL) of sample, diluting to the constant volume of 25 mL and pipetting 1.00 mL of sample digestion solution for determination, the limit of quantification for this method is 100 mg/kg (or 100 mg/L).

Method III -- Inductively coupled plasma atomic emission spectrometry

See GB 5009.268.

Method IV -- Inductively coupled plasma mass spectrometry

See GB 5009.268.

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