GB

NATIONAL STANDARD

OF THE PEOPLE'S REPUBLIC OF CHINA

GB 5009.91-2017

National food safety standard

Determination of potassium and sodium in foods

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Table of contents

Foreword 3

1 Scope 4

2 Principle 4

3 Reagents and materials 4

4 Instruments and equipment 6

5 Analytical procedures 6

6 Expression of analytical results 9

7 Precision 9

8 Others 9

9 Principles 10

10 Reagents and materials 10

11 Instruments and equipment 11

12 Analytical procedures 12

13 Expression of analytical results 12

14 Precision 13

15 Others 13

Appendix A Sample determination reference condition 14

Foreword

This standard replaces the potassium and sodium determination part in GB/T 5009.91-2003 “Determination of potassium and sodium in foods”, GB/T 15402-1994 “Fruits, vegetables and derived products – determination of sodium and potassium”, NY 82.18-1988 “Determination of potassium and sodium in fruit juice”, 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 18932.11-2002 “Method for determination of potassium, phosphorus, iron, calcium, zinc, aluminum, sodium, magnesium, boron, manganese, copper, barium, titanium, vanadium, nickel, cobalt and chromium in honey - Inductively coupled plasma atomic emission spectrometry (ICP-AES method”, GB/T 18932.12-2002 “Method for determination of potassium, sodium, calcium, magnesium, zinc, iron, copper, manganese, chromium, lead and cadmium contents in honey - Atomic absorption spectrometry”, AND NY/T 1653-2008 “Determination for mineral elements in vegetables, fruits, and derived products by ICP-AES method”.

As compared with the above standards, the main changes are as follows:

- CHANGE the standard name into “National food safety standard - Determination of potassium and sodium in foods”;

- MODIFY the flame atomic absorption spectrometry method as the first method, the flame atomic emission spectrometry method as the second method, the inductively coupled plasma emission spectroscopy method as the third method, AND the inductively coupled plasma mass spectrometry as the fourth method;

- MODIFY some part of the sample preparation;

- MODIFY some part of the sample digestion;

- INCREASE the method detection limit and quantitative limit;

- ADD appendix.

National food safety standard

Determination of potassium and sodium in foods

1 Scope

This standard specifies four methods for the determination of potassium and sodium in food: the flame atomic absorption spectrometry, the flame atomic emission spectrometry, the inductively coupled plasma emission spectroscopy, AND the inductively coupled plasma mass spectrometry.

This standard applies to the determination of potassium and sodium in food.

Method I: Flame atomic absorption spectrometry

2 Principle

After digestion, the sample is injected into the atomic absorption spectrometer. After the flame atomization, the potassium and sodium respectively absorb the 766.5 nm and 589.0 nm resonance line. AND within a concentration range, its absorption value is proportional to the contents of potassium and sodium, AND it is quantified through comparison with standard series.

3 Reagents and materials

Unless otherwise specified, the reagents used in this method are excellent grade pure, AND the water is level II 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 Cesium chloride (CsCl).

3.2 Reagent preparation

3.2.1 Mixed acid [perchloric acid + nitric acid (1 + 9)]: TAKE 100 mL of perchloric acid; slowly ADD it into 900 mL of nitric acid; MIX it uniformly.

3.2.2 Nitric acid solution (1 + 99): TAKE 10 mL of nitric acid; slowly ADD it into 990 mL of water; MIX it uniformly.

3.2.3 Cesium chloride solution (50 g/L): DISSOLVE 5.0 g of cesium chloride in water; USE water to dilute it to 100 mL.

3.3 Standard substance

3.3.1 Potassium chloride standard substance (KCl): its purity is greater than 99.99%.

3.3.2 Sodium chloride standard substance (NaCl): its purity is greater than 99.99%.

3.4 Preparation of standard solution

3.4.1 Potassium and sodium standard stock solution (1000 mg/L): PLACE the potassium chloride or sodium chloride in the oven at 110 °C ~ 120 °C to dry it for 2 h. Accurately WEIGH 1.9068 g of potassium chloride or 2.5421 g of sodium chloride; respectively DISSOLVE it into water; TRANSFER it into a 1000 mL volumetric flask; DILUTE it to the mark; MIX it uniformly; STORE it in a polyethylene bottle; PRESERVE it at 4 °C; or otherwise USE the standard solution which had been certified and awarded with the standard substance certificate by the state.

3.4.2 Potassium and sodium standard working solution (100 mg/L): accurately PIPETTE 10.0 mL of potassium or sodium standard stock solution in a 100 mL volumetric flask; USE water to dilute it to the mark; STORE it in a polyethylene bottle; PRESERVE it at 4 °C.

3.4.3 Potassium and sodium standard series working solution: accurately PIPETTE 0 mL, 0.1 mL, 0.5 mL, 1.0 mL, 2.0 mL, and 4.0 mL of potassium standard working solution in 100 mL volumetric flask; ADD 4 mL of cesium chloride solution; USE water to make its volume reach to the mark; MIX it uniformly. The concentration of potassium in this standard series working solution is 0 mg/L, 0.100 mg/L, 0.500 mg/L, 1.00 mg/L, 2.00 mg/L, and 4.00 mg/L, respectively; AND it may, in accordance with the potassium concentration in the actual sample solution, appropriately adjust the concentration range of the standard solution. Accurately PIPETTE 0 mL, 0.5 mL, 1.0 mL, 2.0 mL, 3.0 mL, and 4.0 mL of sodium standard working solution in 100 mL volumetric flask; ADD 4 mL of cesium chloride solution; USE water to make its volume reach to the mark; MIX it uniformly. The concentration of sodium in the standard series working solution is 0 mg/L, 0.500 mg/L, 1.00 mg/L, 2.00 mg/L, 3.00 mg/L, and 4.00 mg/L, respectively; AND it may, in

WEIGH 0.5 g ~ 5 g (accurate to 0.001 g) of sample in the glass OR Teflon pressure digestion tank; as for the samples containing ethanol or carbon dioxide, PLACE it on the heating plate to heat it at low temperature to remove ethanol or carbon dioxide; ADD 10 mL of mixed acid; COVER and PLACE it for 1 h or overnight; PLACE it in the adjustable temperature control electric heating plate or furnace for digestion; if it changes into dark brown, COOL it down and ADD the mixed acid, until white smoke is generated AND the digestion solution is in colorless transparent OR in light yellow; COOL it down; USE water to make its volume reach to 25 mL or 50 mL; MIX it uniformly to prepare for use; and meanwhile MAKE blank test.

5.2.4 Dry digestion method

WEIGH 0.5 g ~ 5 g (accurate to 0.001 g) of sample in the crucible; MAKE it subject to carbonization in the electric furnace at small fire to make it smokeless; PLACE it into the 525 °C ± 25 °C muffle furnace for ashing for 5 h ~ 8 h; COOL it. If there is black carbon particles due to incomplete ashing, COOL it and ADD a small amount of nitric acid to wet it; DRY it on the electric heating plate; TRANSFER it into the muffle furnace to continue ashing to make it become white ash; COOL it to room temperature and TAKE it out; USE nitric acid to dissolve it; USE water to make its volume reach to 25 mL or 50 mL; MIX it uniformly to prepare for use; and meanwhile MAKE blank test.

5.3 Instrument reference conditions

OPTIMIZE the instrument to the best condition, AND the main conditions of the instrument are as shown in Table A.2.

5.4 Production of standard curve

Respectively INJECT the potassium and sodium standard series working solution into the atomic absorption spectrometer; DETERMINE the absorbance value; USE the standard working solution concentration as the abscissa AND the absorbance value as the ordinate; DRAW the standard curve.

5.5 Determination of sample solution

In accordance with the content of the element under determination in the sample solution, USE water to dilute the sample solution to the appropriate concentration if necessary; and ADD a certain amount of cesium chloride solution into the blank solution and the sample final determination solution, to make the concentration of cesium chloride reach to 0.2 %. At the same conditions as for the determination standard curve working solution, INJECT the blank solution and the determination solution into the atomic absorption

$$X = \frac{(\rho - \rho_0) \times V \times f \times 100}{m \times 1\,000} \quad \cdots\cdots (2)$$

Where:

X - The content of the element to be measured in the sample, in milligrams per one hundred milligram or milligram per hundred milliliter (mg/100 g OR mg/100 mL);

ρ - The mass concentration of the element in the determination solution, in milligrams per liter (mg/L);

ρ_0 - The mass concentration of the element in the determination blank solution, in milligrams per liter (mg/L);

V – Volume of sample volume, in milliliters (mL);

f - Dilution factor of sample solution;

100, 1000 - Conversion factor;

m - The mass or volume of the sample, in grams or milliliters (g OR mL).

The calculation result is retained with three significant digits.

14 Precision

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

15 Others

If the sampling amount is 0.5 g and its volume is made to 25 mL, the detection limit of sodium is 0.2 mg/100 g AND the quantification limit is 0.5 mg/100 g; the detection limit of sodium is 0.8 mg/100 g AND the quantification limit is 3 mg/100 g.

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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