NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 5009.267-2020

National food safety standard - Determination of iodine in foods

Issued on: September 11, 2020 **Implemented on: March 11, 2021**

Issued by: National Health Commission and State Administration for Market Regulation

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1 Scope

This standard specifies the determination method of iodine content in food.

The method 1 Inductively coupled plasma mass spectrometry is suitable for the determination of iodine in food.

The method 2 Redox titration is suitable for the determination of iodine in algae and its products.

The method 3 Arsenic-cerium catalytic spectrophotometry is suitable for the determination of iodine in food, vegetables, fruits, beans and their products, milk and their products, meat, fish and eggs.

The method 4 Gas chromatography is applicable to the determination of nutritional fortifier iodine in infant formula foods and dairy products (except infant formula foods for special medical purposes and formula foods for special medical purposes).

Method 1 -- Inductively coupled plasma mass spectrometry (ICP-MS)

2 Principle

The iodine in the specimen is extracted by tetramethylammonium hydroxide solution and measured by inductively coupled plasma mass spectrometer. It is qualitatively determined by the specific mass of iodine element 127 (mass-to-charge ratio, m/z), the mass spectrometry signal intensity ratio between iodine element and internal standard element which is proportional to the concentration of the iodine element is used for quantification, to determine the iodine content in the specimen.

3 Reagents and materials

Unless otherwise specified, the reagents used in this method are all premium-grade pure water; the water is the grade 1 water as specified in GB/T 6682 or the high-purity water used for instrument analysis as specified in GB/T 33087-2016.

3.1 Reagent

3.1.1 25% Tetramethylammonium hydroxide [(CH3)4NOH] aqueous solution.

the English abbreviation name TMAH.

3.3 Standard product

Potassium iodide (KI) or potassium iodate (KIO3). Reference reagent.

3.4.1 Iodine standard stock solution (1000 mg/L). Weigh 0.1685 g of potassium

iodate that has been dried at 180 °C +/- 2 °C to a constant weight. Use water to dissolve and make its volume reach to 100 mL. Or weigh 0.1307 g of potassium iodide which had been dried in a silica gel dryer for 24 hours. Use water to dissolve and dilute it to 100 mL. Store it in a brown bottle. It may also use the iodine standard solution certified nationally and awarded with a certificate of standard material.

3.4.2 Iodine standard intermediate solution (10.0 mg/L). Pipette 1.00 mL of

iodine element standard stock solution. Use diluent to make its volume reach to 100 mL.

3.4.3 Iodine standard solution (100 ug/L). Pipette 1.00 mL of iodine element

standard intermediate solution. Use diluent to make its volume reach to 100 mL.

3.4.6 Internal standard solution. First use water to dilute the internal standard

element standard solution 10 times or 100 times. Then take an appropriate amount of the solution and use the diluent to prepare an internal standard solution of appropriate concentration.

5.1.1.1 Dry sample

For samples of beans, grains, fungi, tea, dried fruits, baked foods, etc., take the edible part; use a high-speed grinder to crush it; stir until uniform. For solid dairy products, protein powders, flour and other homogeneous powder samples, shake it uniformly.

5.1.1.2 Fresh sample

Wash and drain samples of vegetables, fruits, aquatic products, etc., if necessary; homogenize the edible part. For samples of meat and eggs, homogenize the edible part.

5.1.1.3 Quick frozen and canned food

Homogenize the edible part of the thawed quick-frozen food and canned food samples to homogeneity.

5.1.2 Liquid sample

Shake the samples of soft drinks, condiments, etc., uniformly.

5.2 Specimen processing

Weigh 0.2 g ~ 1 g (accurate to 0.001 g) of the specimen in a 50 mL plastic centrifuge tube which is resistant to 110 °C temperature. Add 5 mL of extraction solution. Vortex for 1 min, to make the sample fully dispersed.

5.3.1 Reference working conditions for instrument operation

Radio frequency power 1550 W; plasma gas flow rate 15 L/min; carrier gas flow rate 0.80 L/min ~ 0.90 L/min; auxiliary gas flow rate 0.30 L/min ~ 0.40 L/min;

pump speed during analysis.

5.3.2 Measurement reference conditions

After the tuning instrument meets the measurement requirements, edit the measurement method and select the iodine isotope (127I) and the internal standard tellurium isotope (125Te, 130Te) or 103Rh or 115In or 185Re.

5.4 Preparation of standard curve

Inject iodine standard solution into ICP-MS. Measure the signal response value of iodine element and internal standard element. Take the mass concentration of iodine element as the abscissa, the ratio of the response signal value of iodine element and the selected internal standard element as the ordinate, to draw standard curve.

5.5 Measurement of sample solution

Inject the blank and sample solution into the inductively coupled plasma mass spectrometer, respectively. Measure the signal response values of the iodine element and the selected internal standard element. Calculate the ratio of the response signal value of the iodine element and the selected internal standard element.

6 Expression of analysis results

The iodine content in the specimen is calculated according to formula (1).

7 Precision

When the iodine content in the sample is greater than 1 mg/kg, the absolute difference between the two independent determination results obtained under repeatability conditions shall not exceed 10% of the arithmetic mean. When it is less than or equal to 1 mg/kg and greater than 0.1 mg/kg, the absolute difference between two independent determination results obtained under repeatability conditions shall not exceed 15% of the arithmetic mean.

8 Others

Calculated with a sampling volume of 0.5 g and a constant volume to 50 mL, the detection limit of the method is 0.01 mg/kg; the limit of quantification is 0.03 mg/kg.