National Standards of People's Republic of China

GB 5009.123-2014

National Food Safety Standard

Determination of Chromium in Foods

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Issued by National Health and Family Planning Commission

Foreword

This standard will replace GB/T 5009.123-2003 “ Determination of Chromium in Foods”.

Compared to GB/T 5009.123-2003, this standard maintains the following major changes:

- Name of the standard is modified into “ National Standard for Food Safety - Determination of Chromium in Foods”;
- Specimen pre-treatment is increased with microwave digestion and wet digestion;
- Limit of Quantitation (LOQ) is added;
- Ammonium dihydrogen phosphate instead of ammonium phosphate is used as matrix modifier;
- Method Two Oscillopolarography Law is deleted.

National Standard for Food Safety

Determination of Chromium in Foods

1. Scope

This standard specifies the determination of chromium in foods by graphite furnace atomic absorption spectrometric method.

This standard applies to determination of chromium content in various types of foods.

2. Principle

After the specimen digests, the graphite furnace atomic absorption spectrometric method will be adopted to measure the absorbance at 357.9 nm. Compare the absorbance to limit of qualification of the standard series solution under the given concentration range.

3. Reagents and materials

Note: except otherwise specified, all reagents used in this method are guaranteed pure reagents and water are Class II water specified in GB / T6682.

3.1 Reagents

3.1.1 Nitric acid (HNO_3).

3.1.2 Perchloric acid (HCIO4).

3.1.3 Ammonium dihydrogen phosphate (NH4 H2PO4)

3.2 Reagent compounding

3.2.1 Nitric acid solution (5 + 95): measure and take 50 ml of nitric acid, slowly pour it into 950 ml of water, and mix it well.

3.2.2 Nitric acid solution (1 + 1): measure and take 250 ml of nitric acid, slowly pour it into 250 ml of water, and mix it well.

3.2.3 Ammonium dihydrogen phosphate solution (20 g/l): weigh and take 2.0 g of ammonium dihydrogen phosphate, dissolve it into water and dilute it to 100 ml, and mix it well.

3.3 Standard products

Potassium dichromate ($K_2Cr_2O_7$): purity> 99.5%, or reference materials certified by and granted with reference materials certificate by the State.

3.4 Formulation of standard solution

3.4.1 Chromium standard stock solution: weigh 1.4315 g (accurate to 0.0001 g) of the reference material potassium dichromate (110℃ baked for 2h), dissolve it into water and transfer it into 500 ml volumetric flask, dilute it with nitric acid solution (5+ 95) to the mark, and mix it well. This solution contains 1.000 mg of chromium per milliliter. Chromium standard stock solution certified by the State and granted with reference material certificate shall be purchased.

3.4.2 Chromium standard use solution: dilute chromium standard use solution by nitric acid solution (5 + 95) progressively till the content of chromium per ml is 100 ng.

3.4.3 Preparation of standard series solutions: imbibe and take respectively 0 ml, 0.500 ml, 1.00 ml, 2.00 ml, 3.00 ml, and 4.00 ml of chromium standard use solution (100 ng/ml) and tranfer it to 25 ml volumetric flask; dilute it with nitric acid solution (5 + 95) to the mark, and mix it well. Chromium content per milliliter in each volumetric flask shall be 0 ng, 2.00 ng, 4.00 ng, 8.00 ng, 12.0 ng, and 16.0 ng respectively. Or, graphite furnace automatic specimenr might be adopted for automatic preparation.

4. Equipment and facilities

Note: all glassware applied shall be soaked in nitric acid solution (1 + 4) for 24h or above, washed with water repeatedly, and finally washed and rinsed with deionized water.

4.1 Atomic absorption spectrometer, equipped with graphite furnace atomizer, and attached with chromium hollow cathode lamp.

4.2 Microwave digestion system, equipped with inner digestion tank.

4.3 Adjustable electric furnace.

4.4 Adjustable heating plate.

4.5 Pressure digestion device, equipped with inner digestion tank.

4.6 Muffle furnace.

4.7 Constant temperature drying oven.

4.8 Electronic balance: a sensor volume of 0. 1 mg and 1 mg.

5. Analytical procedures

5.1 Pretreatment of specimen

5.1.1 Crush and load grains, beans and other foodstaffs into a clean container after foreign matters are removed, and use them as specimen. Seal and label clearly; specimens shall be stored at room temperature.

5.1.2 Fresh specimens with higher content of water such as vegetables, fruits, fish, meat and eggs shall be directly refined into homogenized solution, loaded into clean container and used as specimen; seal and label clearly; specimen shall be stored in refrigerator freezer.

5.2 Specimen digestion

5.2.1 Microwave digestion

Accurately weigh and take 0.2g~0.6g (accurate to 0.001g) of specimen and transfer it into microwave digestion tank; add 5 ml nitric acid; digest specimen as per microwave digestion procedure (please refer to A.1 for microwave digestion conditions); take out the digestion tank after it cools down; clear up acid to 0.5 ml~1.0 ml on electric hot plate under the temperature of 140℃~160℃. After digestion tank cools down, transfer the digestion solution to 10 ml volumetric flask, wash digestion tank by a small amount of water for 2~3 times; combined the washing liquid, dilute it by water to the mark; conduct reagent blank test at the same time.

5.2.2 Wet digestion

Weigh and take 0.5 g~3 g (accurate to 0.001 g) of specimen and transfer it into digestive tract; add 10 ml of nitric acid and 0.5 ml of perchloric acid; digest on the adjustable electric furnace (reference conditions: 120℃, kept for 0.5h~1h, heated up to 180℃ for 2h~4h, and heated up to 200℃~220℃). If the digestion liquid is brown in color, add nitric acid into it until white smoke rises; digestion liquid shall be colorless, transparent or slightly yellowish; take out the digestive tract, cool it down and dilute it by water to 10 ml. At the same conduct the blank test for the reagent.

5.2.3 High pressure digestion

Accurately weigh and take 0.3 g~1 g (accurate to 0.001 g) of specimen and transfer it into digestion tank; add 5 ml of nitric acid; replace the inner cover; tighten the stainless steel jacket and put it into the constant temperature oven; maintain it for 4h~5h at 140℃~160℃. Cool it down naturally to room temperature within the constant temperature oven; slowly loosen the outer tank and take out the inner digestion tank; place it onto the adjustable electric hot plate and clear up acid to 0.5 ml~1.0 ml at 140℃~160℃. After cooling, transfer the digestion liquid into a 10 ml volumetric flask, and wash the inner tank by a small amount of water for 2~3 times. Combine the wash lotion into the volumetric flask and dilute it by water to the mark; at the same, conduct the blank test for the reagent.

5.2.4 Dry ashing

Weigh and take 0.5 g~3 g (accurate to 0.001 g) of specimen and transfer it into a crucible; simmer till it is carbonized and smokeless; transfer it into a muffle furnace; maintain it for 3h~4h at 550℃. Take it out and cool down. For specimen that is not fully ashed, add a few drops of nitric acid, simmer, and evaporate carefully; then transfer it into high temperature furnace of 550℃ and continue ashing treatment for 1h~2h till the specimen is lime-like; take it out from the high-temperature furnace and cool down; dissolve it by nitric acid solution (1 + 1) and dilute it with water to 10 ml; at the same time conduct the blank test for the reagent.

5.3 Determination

5.3.1 Test conditions of instrument

Calibrate to the optimal state as per performance of the respective instrument. For reference conditions, please refer to A.2.

5.3.2 Standard curve mapping

Take respectively 10 µl (you can select the best injection volume as per the instrument applied) of standard series solution working solution bottom up in terms of concentration, inject it into graphite tube, and measure its absorbance after it is being atomized; take concentration as the horizontal axis and absorbance vertical axis and map the standard curve.

5.3.3 Specimen determination

Under the same experimental conditions under which the standard solution is determined, take respectively 10 µl of the blank solution and specimen solution (the best injection volume might be subject to the instrument applied); inject them into graphite tube; measure its absorbance value after it is being atomized; compare to the limit of quantification of the standard series solution.

Inject 5 µl (the best injection volume can be subject to the instrument applied) of ammonium dihydrogen phosphate solution (20.0 g/l) into the interferred specimen (preparation process of the standard series solution specified in 5.3.3 shall be followed).

6. Statement of analysis result

For calculation of chromium content in the specimen, please refer to Formula (1):

$$X = \frac{(c - c_0) \times V}{m \times 1\ 000} \qquad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (1)$$

In the formula:

X- chromium content in the specimen, in milligram per kilogram (mg/kg);

c- determination of chromium content in the specimen solution, in nanogram per milliliter (ng/ml);

c_0- chromium content in blank solution, in nanogram per milliliter (ng/ml);

V- total constant volume of specimen digestion solution, in milliliter (ml);

m- volume of specimen weighed, in gram (g);

1 000 - conversion coefficient.

When the analysis result ≥1 mg/kg, keep a three-digit valid number; when the analysis result < 1mg/kg, keep a two-digit valid number.

7. Precision

Absolute difference between two independent determination results obtained under the repeatability conditions shall not exceed 20% of their arithmetic mean.

8. Others

Calculate by 0.5 g of the specimen weighed and dilute it to 10 ml; the detection limit in such case shall be 0.01 mg/kg, and the limit of quantification 0.03 mg/kg.

Appendix A

Reference Conditions for Specimen Determination

A.2 Please refer to Table A for reference conditions for microwave digestion

Table A.1 Reference conditions for microwave digestion

Step	Power (1,200w), change/%	Set temperature / ℃	Warm-up time /min	Holding time /min
1	0~80	120	5	5
2	0~80	160	5	10
3	0~80	180	5	10

A.3 For reference conditions of graphite furnace atomic absorption spectrometry, please refer to Table A.2.

Table A.2 Reference conditions of graphite furnace atomic absorption spectrometry

Element	Wavelength/nm	Slits /nm	Lamp current /mA	Drying /(℃/s)	Ashing (℃/s)	Atomization (℃/s)
Chromium	357.9	0.2	5~7	(85~120)/(40~50)	900/(20~30)	2,700(4~5)