GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 5009.182-2017

National food safety standard -

Determination of aluminum in foods

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Issued by: National Health and Family Planning Commission of the PRC;
China Food and Drug Administration.

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Foreword

For the determination method of aluminum, this standard replaces GB/T 5009.182-2003 “Determination of aluminum in flour products”, GB/T 23374-2009 “Determination of aluminium in foods - Inductively coupled plasma mass spectrometry”, GB/T 18932.11-2002 “Method for the determination of potassium, phosphorus, iron, calcium, zinc, aluminium, sodium, magnesium, boron, manganese, copper, barium, titanium, vanadium, nickel, cobalt, chromium contents in honey. Inductively coupled plasma atomic emission spectrometric method (ICP-AES)”, SN/T 2208-2008 “Determination of sodium, magnesium, aluminium, calcium, chromium, iron, nickel, copper, zinc, arsenic, strontium, molybdenum, cadmium, lead, mercury, selenium, in aquatic products - Microwave digestion - ICP/MS method ".

As compared with GB/T 5009.182-2003, the main changes of this standard are as follows:

---The standard name is changed to “National food safety standard - Determination of aluminium in foods";

---Improve method I spectrophotometry;

---Add inductively coupled plasma mass spectrometry as method II;

---Add inductively coupled plasma emission spectrometry as method III;

---Add graphite furnace atomic absorption spectrometry as method IV.

National food safety standard -

Determination of aluminum in foods

1 Scope

This standard specifies some methods for determination of aluminum content in foods, such as spectrophotometry, inductively coupled plasma mass spectrometry, inductively coupled plasma emission spectrometry and graphite furnace atomic absorption spectrometry.

Method I in this standard applies to the detection of aluminum in foods containing aluminum-based food additives. Method II, method III and method IV are applicable to the detection of aluminum in foods.

Method I: Spectrophotometry

2 Principles

After the sample is treated, it is in ethylenediamine-HCl buffer solution (pH 6.7-7.0), with the existence of Triton X-100 and cetylpyridinium bromide (CPB), trivalence aluminum ion reacts with chrome azurol S to generate blue-green quaternary micelle. The absorbance value is measured at a wavelength of 620 nm and compared with the standard series for quantitation.

3 Reagents and materials

Unless otherwise indicated, the reagents used in this method are analytical pure, the experimental water is the grade-3 water specified in GB/T 6682.

3.1 Reagents

3.1.1 Nitric acid (HNO_3): guaranteed reagent.

3.1.2 Sulfuric acid (H_2SO_4): guaranteed reagent.

3.1.3 Hydrochloric acid (HCl): guaranteed reagent.

3.1.4 Ammonium hydroxide ($NH_3 \cdot H_2O$): guaranteed reagent.

3.1.5 Anhydrous ethanol (C_2H_6O): guaranteed reagent.

3.1.6 P-nitrophenol ($C_6H_5NO_3$).

3.1.7 Chrome azurol S ($C_{23}H_{13}O_9SCl_2Na_3$).

3.1.8 Ethylenediamine ($C_2H_8N_2$).

3.1.9 Polyethylene glycol octyl phenyl ether (Triton X-100).

3.1.10 Cetylpyridinium bromide (CPB, $C_{21}H_{38}BrN$).

3.1.11 Ascorbic acid ($C_6H_8O_6$).

3.2 Reagent preparation

3.2.1 Hydrochloric acid solution (1+1): MEASURE 50 mL of hydrochloric acid; MIX it uniformly with 50 mL of water.

3.2.2 Sulfuric acid solution (1%): PIPETTE 1 mL of sulfuric acid; POUR slowly into 80 mL of water; USE water to dilute it to 100 ml after cooling; MIX it uniformly.

3.2.3 P-nitrophenol ethanol solution (1 g/L): WEIGH 0.1 g of p-nitrophenol; DISSOLVE it in 100 mL of anhydrous ethanol; MIX it uniformly.

3.2.4 Nitric acid solution (5%): MEASURE 5 mL of nitric acid; ADD water to dilute it to 100 mL; MIX it uniformly.

3.2.5 Nitric acid solution (2.5%): MEASURE 2.5 mL of nitric acid; ADD water to dilute it to 100 mL; MIX it uniformly.

3.2.6 Ammonium hydroxide solution (1+1): MEASURE 10 mL of ammonium hydroxide; POUR into 10 mL of water; MIX it uniformly.

3.2.7 Nitric acid solution (2+98): MEASURE 2 mL of nitric acid; MIX it uniformly with 98 mL of water.

3.2.8 Ethanol solution (1+1): MEASURE 50 mL of anhydrous ethanol; DISSOLVE it in 50 mL of water; MIX it uniformly.

3.2.9 Chrome azurol S solution (1 g/L): WEIGH 0.1 g of chrome azurol S; DISSOLVE it in 100 mL of ethanol solution (1+1); MIX it uniformly.

3.2.10 TritonX-100 solution (3%): PIPETTE 3 mL of TritonX-100; PLACE it into 100 mL volumetric flask; ADD water to dilute it to the mark; MIX it uniformly.

3.2.11 CPB solution (3 g/L): WEIGH 0.3 g of CPB; DISSOLVE it in 15 mL of anhydrous ethanol; ADD water to dilute it to 100 mL; MIX it uniformly.

5 Analytical procedures

5.1 Sample Preparation

In the process of sampling and sample preparation, pay attention not to contaminate the sample; avoid using aluminum-containing appliances.

After crushing the samples of flour products, bean products, shrimp flavorings and bakery products uniformly, TAKE out 30 g of sample; PLACE it into constant-temperature oven at 85°C for 4h.

5.2 Sample digestion

WEIGH 0.2 g ~ 3 g of sample (accurate to 0.001g) or accurately MOVE 0.500 mL ~ 5.00 mL of liquid sample into hard glass digestion tube or conical flask; ADD 10 mL of nitric acid and 0.5 mL of sulfuric acid; HEAT it on adjustable temperature-control electric furnace or hot plate, reference conditions: heat it at 100°C for 1h; rise to 150°C and heat it for 1h; rise to 180°C and heat it for 2h; then rise to 200°C. If it becomes brownish black, ADD nitric acid again to digest; until the tube mouth emits white smoke, digestive juice is colorless and transparent or slightly yellow. TAKE it out; after cooling, USE water to transfer and dilute it to 50 mL (V_1) volumetric flask; MIX it uniformly for spare-use. Meanwhile, DO a reagent blank test.

5.3 Color reaction and colorimetric determination

Respectively PIPETTE 1.00 mL (V_2) of sample digestive juice and blank solution; PLACE them into 25 mL plugged colorimetric tube; ADD water to 10 mL. In addition, TAKE seven 25 mL plugged colorimetric tubes; ADD respectively aluminum standard working solution 0 mL, 0.500 mL, 1.00 mL, 2.00 mL, 3.00 mL, 4.00 mL and 5.00 mL (in the series standard solution, the mass of aluminum is 0 μg, 0.500 μg, 1.00 μg, 2.00 μg, 3.00 μg, 4.00 μg, 5.00 μg respectively); ADD 1 mL of sulfuric acid solution (1%) in each tube successively; ADD water to 10 mL.

ADD dropwise 1 drop of p-nitrophenol ethanol solution (1 g/L) into standard tube, sample tube and reagent blank tube; MIX it uniformly; ADD dropwise ammonium hydroxide solution (1+1) until the solution is light yellow; ADD dropwise nitric acid solution (2.5%) until the yellow just disappears; ADD again 1 mL of nitric acid solution; ADD 1 mL of ascorbic acid solution (10 g/L); after mixing it uniformly, ADD 3 mL of chrome azurol S solution (1 g/L); after mixing it uniformly, ADD 1 mL of TritonX-100 solution (3%), 3 mL of CPB solution (3 g/L), 3 mL of ethylenediamine - hydrochloric acid buffer solution; ADD water to dilute it to 25.0 mL; MIX it uniformly; PLACE it for 40min.

10.4 Standard solution preparation

10.4.1 Aluminum standard intermediate solution (100 mg/L): Accurately PIPETTE 1.00 mL of aluminum standard solution (1000 mg/L) into 10 mL volumetric flask; ADD nitric acid solution (5+95) to dilute it to the mark; MIX it uniformly.

10.4.2 Aluminum standard working solution (1.00 mg/L): Accurately PIPETTE 1.00 mL of aluminum standard intermediate solution (100 mg/L) into 100 mL volumetric flask; USE nitric acid solution (5 + 95) to dilute it to the mark; after mixing it uniformly, PIPETTE accurately 1.00 mL from the solution; PLACE it into 100 mL volumetric flask; USE water to dilute it to the mark; MIX it uniformly.

10.4.3 Aluminum standard series solution: PIPETTE respectively aluminum standard solution (1.00 mg / L) 0 mL, 2.50 mL, 5.00 mL, 10.0 mL, 15.0 mL and 20.0 mL into 100 mL volumetric flask; ADD nitric acid solution (1+99) to the mark; MIX it uniformly. The mass concentration of the aluminum standard solution is 0 μg/L, 25.0 μg/L, 50.0 μg/L, 100 μg/L, 150 μg/L and 200 μg/L respectively.

11 Instrument and equipment

Note: All glassware and digestion pots must be soaked in nitric acid solution (1+5) for more than 24h, washed repeatedly with tap water, finally rinsed with water. Dry it by airing before use.

11.1 Graphite Furnace Atomic Absorption Spectrometer: Attached aluminum hollow cathode lamp.

11.2 Balance: sensitivity is 1 mg.

11.3 Adjustable temperature-control electric furnace.

11.4 Adjustable hot plate.

11.5 Microwave digestion instrument: equipped with Teflon digestion inner-tank.

11.6 Pressure digestion pot: equipped with Teflon digestion inner-tank.

11.7 Constant-temperature oven

12 Analytical procedures

12.1 Sample preparation

temperature, OPEN the digestion tank; CATCH-acid to dry on electric hot plate; when it is reduced to room temperature, USE a small amount of water to wash the digestion tank for 3~4 times; COMBINE washing liquid in 25 mL volumetric flask; Use water to dilute it to the mark; MIX it uniformly for spare-use. Meanwhile, DO a sample blank test.

12.2.3 Pressure-tank digestion

WEIGH 0.2 g ~ 1 g of solid sample (accurate to 0.001 g) or accurately MOVE 0.500 mL ~ 5.00 mL of liquid sample into pressure digestion inner-tank; ADD 5 mL~ 8mL of nitric acid; COVER inner-cap; TIGHTEN outer-sheath; PLACE it into constant-temperature oven; after digestion tank is cooled to room temperature, OPEN the pressure digestion tank; TAKE out inner-tank; CATCH-acid to dry on electric hot plate; when it is reduced to room temperature, USE a small amount of water to wash the digestion tank for 3~4 times; COMBINE washing liquid in 25 mL volumetric flask; Use water to dilute it to the mark; MIX it uniformly for spare-use. Meanwhile, DO a sample blank test. (specific digestion conditions refer to Appendix B).

12.3 Determination

12.3.1 Instrument's test conditions

Adjust to optimum state according to performance of each instrument. The reference conditions are a wavelength of 257.4 nm, a slit of 0.5 nm, a lamp current of 10 mA ~ 15 mA, a drying temperature of 85° C. ~ 120° C. for 30 s; an ashing temperature of 1000° C. ~ 1200° C. for 15 s to 20 s; an atomization temperature of 2750° C. for 4s ~ 5s; internal gas flow 0.3 L/min, the injection volume 10 μL, stop gasification during atomization.

12.3.2 Drawing of standard curve

LEAD 10 μL of standard series solution (select the best injection volume according to instrumentation) into graphite tube from low to high according to mass concentration; MEASURE absorbance value after atomization. With the mass concentration as abscissa AND absorbance value as vertical axis, DRAW a standard curve.

12.3.3 Determination of sample solution

According to the best injection volume of instrument, LEAD respectively sample digestive juice and blank solution of appropriate volume into graphite furnace to measure absorbance value. The mass concentration of aluminum in the sample digestive juice is obtained from the standard curve.

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