



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB 5009.90-2016

National food safety standard - Determination of iron in foods

食品安全国家标准 食品中铁的测定

(English Translation)

Issued on: December 23, 2016

Implemented on: June 23, 2017

Issued by: National Health and Family Planning Commission of the PRC;

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Foreword

This standard replaces methods for the determination of iron content in 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 child"; GB/T 23375-2009 "Determination of copper, iron, zinc, calcium, magnesium and phosphorus content in vegetables and derived products"; GB/T 5009.90-2003 "Determination of iron, magnesium and manganese in foods"; 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 18932.12-2002 "Method for the determination of potassium, sodium, calcium, magnesium, zinc, iron, copper, manganese, chromium, lead, cadmium contents in honey - Atomic absorption spectrometry"; GB/T 9695.3-2009 "Meat and meat products - Determination of iron content"; NY/T 1201-2006 "Determination of copper, iron and zinc content in vegetables and derived products".

As compared with GB/T 5009.90-2003, the main changes of this standard are as follows.

- Standard name is changed to "National food safety standard -Determination of iron in foods";
- Add microwave digestion, pressure tank digestion and dry digestion;
- Add the inductively coupled plasma emission spectrometry;

--- Add the inductively coupled plasma mass spectrometry;

--- Delete spectrophotometry.

National Food Safety Standard -Determination of iron in foods

1 Scope

This standard specifies the determination of iron content in foods by flame atomic absorption spectrometry, inductively coupled plasma emission spectrometry and inductively coupled plasma mass spectrometry.

This standard applies to the determination of iron content in foods.

Method I. Flame atomic absorption spectrometry

2 Principles

After digestion, the sample is atomized by atomic absorption and the absorbance value is measured at 248.3 nm. Within a certain concentration range, iron absorbance value is directly proportional to iron content; compares with the standard series for quantitation.

3 Reagents and materials

Unless otherwise indicated, the reagents used in this method are guaranteed reagent, the water is the grade-2 water specified in GB/T 6682.

3.1 Reagents

3.1.1

Nitric acid (HNO₃).

3.2 Reagent preparation

3.2.1

Nitric acid solution (5+95). MEASURE 50 mL of nitric acid; POUR into 950mL of water; MIX it uniformly.

3.3 Standards

Ferric ammonium sulfate [NH₄Fe(SO₄)₂·12H₂O, CAS number 7783-83-7].

Purity > 99.99%. Or iron standard solution with a certain concentration which is identified by national authentication and awarded a reference material certificate.

3.4.1 Iron standard stock solution (1000 mg/L). WEIGH accurately 0.8631 g

(accurate to 0.0001 g) of ammonium ferric sulfate; ADD water to dissolve it;

ADD 1.00 mL of sulfuric acid solution (1+3);

3.4.2 Iron standard intermediate solution (100 mg/L). PIPETTE accurately

10mL of iron standard stock solution (1000 mg/L) into a 100mL volumetric flask;

ADD nitric acid solution (5+95) to the mark; MIX it uniformly. The mass concentration of the iron solution is 100 mg/L.

4 Instrument and equipment

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

4.1 Atomic Absorption Spectrometer. equipped with flame atomizer, iron hollow cathode lamp.

4.5 Adjustable hot plate.

4.6 Pressure digestion tank. equipped with Teflon digestion inner-tank.

4.7 Constant temperature oven.

4.8 Muffle furnace.

5.1 Sample preparation

Note: During sampling and preparation, AVOID sample contamination.

5.1.1 Samples of food and beans

Remove debris of samples; crush; store it in plastic bottles.

5.2.1 Wet digestion

Accurately WEIGH 0.5 g ~ 3 g of solid sample (accurate to 0.001 g) or accurately MOVE 1.00 mL ~ 5.00 mL of liquid sample into graduated digestive tube; ADD 10 mL of nitric acid and 0.5 mL of perchloric acid;

5.2.2 Microwave digestion

Accurately WEIGH 0.2 g ~ 0.8 g of solid sample (accurate to 0.001 g) or accurately MOVE 1.00 mL ~ 3.00 mL of liquid sample into microwave digestion tank; ADD 5mL of nitric acid;

5.2.4 Dry digestion

Accurately WEIGH 0.5 g ~ 3 g of solid sample (accurate to 0.001 g) or accurately MOVE 2.00 mL ~ 5.00 mL of liquid sample into a crucible; HEAT it in soft fire; CARBONIZE to smokeless; TRANSFER it into a muffle furnace; ASH at 550°C for 3h ~ 4h. Take out after cooling; for sample that is not completely ashed, ADD a few drops of nitric acid; HEAT in soft fire; EVAPORATE carefully;

TRANSFER it into a muffle furnace at 550°C; CONTINUE ashing for 1h ~ 2h until the sample is in white-gray; TAKE out after cooling; DISSOLVE it with appropriate amount of nitric acid solution (1+1); TRANSFER it into a 25 mL volumetric flask. USE a small amount of water to wash the inner tank and the inner caps for 2~3 times; COMBINE washing liquid in the volumetric flask; Use water to dilute it to the mark. Meanwhile. DO a sample blank test.

5.3.1 Instrument's test conditions

Reference conditions are shown in Table B.1.

6 Analysis results expression

The iron content in the sample is calculated in accordance with the equation (1).

7 Precision

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

8 Others

When the sampling weight is 0.5 g (or 0.5 mL) and the constant-volume is 25 mL, the method detection limit is 0.75 mg/kg (or 0.75 mg/L) and the quantitative limit is 2.5 mg/kg (or 2.5 mg/L).

Appendix A

Microwave digestion temperature-rise program Microwave digestion temperature-rise program is as shown in Table A.1.

Appendix B

Reference conditions of flame atomic absorption spectrometry Reference conditions of flame atomic absorption spectrometry are as shown in

Table B.1.