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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

- Released on 2016-12-23
- Implemented on 2017-06-23
- Issued by NHFPC & CFDA

Disclaimer: The English version is an unofficial translation of the original in Chinese for information and reference purposes only. In case of a discrepancy the Chinese original standard will prevail.

Foreword

This standard replaces 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 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” and NY/T 1201-2006 “Determination of copper, iron and zinc content in vegetables and derived products”.

Compared with GB/T 5009.90-2003, major changes of this standard are as follows:

— The standard name has been modified to “National food safety standard - Determination of iron in foods”;

— The microwave digestion, pressure tank digestion and dry digestion have been added;

— The inductively coupled plasma emission spectrometry has been added;

— The inductively coupled plasma mass spectrometry has been added;

— The spectrophotometry has been deleted.

National Food Safety Standard

Determination of Iron in Foods

1. Scope

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

Method I Flame Atomic Absorption Spectrometry

2. Principle

After being digested, the test sample is atomized by atomic absorption flame and its absorbance value is determined at the wavelength of 248.3 nm. In a certain concentration range, the absorbance of iron is proportional to the iron content. And then the quantitative analysis is performed by comparing with standard series.

3. Reagents and Materials

Notes: Unless otherwise specified, the reagents in this method all refer to guaranteed reagents and the water refers to Grade 2 water 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 Sulfuric acid (H_2SO_4)

3.2 Preparation of reagents

3.2.1 Nitric acid solution (5+95): Measure 50 mL of nitric acid, pour it into 950 mL of water and then mix absolutely.

3.2.2 Nitric acid solution (1+1): Measure 250 mL of nitric acid, pour it into 250 mL of water and then mix absolutely.

3.2.3 Sulfuric acid solution (1+3): Measure 50 mL of sulfuric acid, slowly pour it into 150 mL of water and then mix absolutely.

3.3 Standard substance

Ammonium ferric sulfate [$NH_4Fe(SO_4)_2 \cdot 12H_2O$, CAS number 7783-83-7]: purity>99.99%, or a certain concentration of iron standard solution certified by the state and granted a certificate of Reference Material.

3.4 Preparation of standard solutions

3.4.1 Iron standard stock solution (1000 mg/L): Accurately weigh 0.8631g (accurate to 0.0001 g) of ammonium ferric sulfate, add water to dissolve it, add 1.00 mL of sulfuric acid solution (1+3), transfer into a 100mL volumetric flask and dilute to volume with water. Mix absolutely. The mass concentration of this iron solution is 1000 mg/L.

3.4.2 Iron standard intermediate solution (100 mg/L): Accurately pipette 10 mL of iron standard stock solution (1000 mg/L) into a 100mL volumetric flask, dilute to volume with nitric acid solution (5+95) and mix absolutely. The mass concentration of this iron solution is 100 mg/L.

3.4.3 Iron standard series solutions: Accurately pipette 0 mL, 0.500 mL, 1.00 mL, 2.00 mL, 4.00 mL and 6.00 mL of iron standard intermediate solution (100 mg/L) respectively into six 100mL volumetric flasks, dilute to

volume with nitric acid solution (5+95) and mix absolutely. The mass concentration of iron in these iron standard series solutions is 0 mg/L, 0.500 mg/L, 1.00 mg/L, 2.00 mg/L, 4.00 mg/L and 6.00 mg/L, respectively.

Notes: The specific concentration of iron in the standard solution series can be determined based on the sensitivity of the apparatus and the actual content of iron in the sample.

4. Apparatus and Equipment

Notes: All the glass wares and the PTFE inner tank in the digestion tank shall be soaked in nitric acid (1+5) overnight and washed with tap water repeatedly, and finally they shall be cleaned with water.

4.1 Atomic absorption spectrometer: equipped with flame atomizer and iron hollow cathode lamp
4.2 Analytical balance: with the sensitivity of 0.1 mg and 1 mg
4.3 Microwave digestion system: equipped with a PTFE inner tank in the digestion tank
4.4 Adjustable electrothermal furnace
4.5 Adjustable electric hot plate
4.6 Pressure digestion tank: equipped with a PTFE inner tank in the digestion tank
4.7 Constant temperature drying oven
4.8 Muffle furnace

5. Analysis Procedures

5.1 Preparation of test sample
Notes: Samples should be protected from contamination during sampling and preparation.
5.1.1 Sample of grain and bean
After the foreign materials are removed from the sample, smash the sample and store in a plastic bottle.
5.1.2 Sample of vegetable, fruit, fish and meat, etc.
Wash the sample with water, dry in the air, take the edible portion, make it into homogenate and store in a plastic bottle.
5.1.3 For any liquid sample of beverage, alcohol, vinegar, soybean sauce, edible vegetable oil and liquid milk, etc., shake the sample absolutely.

5.2 Digestion of test sample
5.2.1 Wet digestion
Accurately weigh 0.5 g~3 g (accurate to 0.001 g) of solid test sample or accurately pipette 1.00 mL~5.00 mL of liquid test sample in a scaled digestive tube, add 10 mL of nitric acid and 0.5 mL of perchloric acid and digest on the adjustable electrothermal furnace (conditions for reference: digest at 120 ℃ for 0.5 h~1 h, rise to 180 ℃ and digest for 2 h~4 h and then rise to 200℃~220 ℃). If the digestion solution is dark brown, add more nitric acid and continue the digestion until white smoke appears and the digestion solution turns colorless and transparent or yellowish. Remove the digestive tube, allow it to cool and then transfer the digestion solution into a 25mL volumetric flask; Wash 2 or3 times with a small amount of water, combine the washings into the volumetric flask and then dilute to volume with water and mix absolutely until ready for use. Conduct the blank test without adding the test sample at the same time. As mentioned above, a conical flask may be applied alternatively on the adjustable electric hot plate for wet digestion.
5.2.2 Microwave digestion
Accurately weigh 0.2 g~0.8 g (accurate to 0.001 g) of solid test sample or accurately transfer 1.00mL~3.00mL of liquid test sample in the microwave digestion tank, add 5 mL of nitric acid and then digest the test sample according to the operation procedures of microwave digestion (refer to Table A.1 for the digestion conditions). Remove the digestion tank after cooling and then expel acid to about 1.0 mL on the electric hot plate at 140 ℃~160 ℃. After cooling, transfer the digestion solution into a 25mL volumetric flask, wash the inner tank and inner lid 2 or

3 times with a small amount of water, combine the washings in the volumetric flask and then dilute to volume with water and mix absolutely until ready for use. Conduct the blank test without adding the test sample at the sametime.

5.2.3 Pressure tank digestion

Accurately weigh 0.3 g~2 g (accurate to 0.001 g) of solid test sample or accurately pipette 2.00 mL~5.00 mL of liquid test sample in the inner tank of the digestion tank and add 5 mL of nitric acid. Cover the inner lid well and tighten the outer stainless-steel sleeve, place in the constant temperature drying oven and maintain for 4 h~5 h at 140 ℃~160 ℃. After cooling, loosen the outer tank slowly, remove the inner tank of the digestion tank, place it on the adjustable electric hot plate and then expel acid to about 1.0 mL at 140 ℃~160 ℃. After cooling, transfer the digestion solution into a 25mL volumetric flask, wash the inner tank and inner lid 2~3 times with a little water, combine the washings in the volumetric flask and then dilute to volume with water and mix absolutely for use later. Conduct the blank test without adding the test sample at the sametime.

5.2.4 Dry digestion

Accurately weigh 0.5 g~3 g (accurate to 0.001 g) of solid test sample or accurately pipette 2.00 mL~5.00 mL of liquid test sample in the crucible, heat over low fire, conduct carbonization treatment until no smoke appears, transfer into the muffle furnace and conduct ashing treatment at 550 ℃ for 3 h~4 h. Cool down and then remove it from the muffle furnace. For any test sample undergoing incomplete ashing treatment, add several drops of nitric acid, heat over low fire, carefully evaporate to dryness and then transfer into the muffle furnace and continue ashing for 1 h~2 h at 550 ℃ until the test sample is present in the form of white ash. Cool down and then remove it from the muffle furnace. Dissolve it with an appropriate amount of nitric acid solution (1+1), transfer into a 25mL volumetric flask, wash the inner tank and inner lid 2~3 times with a small amount of water,combine the washings in the volumetric flask and then dilute to volume with water. Conduct the blank test without adding the test sample at the sametime.

5.3 Determination

5.3.1 Test conditions of instrument

See Table B.1 for the conditions for reference.

5.3.2 Plotting of standard curve

The standard series of working fluid was introduced into the flame atomizer in the order of mass concentration from low to high, and the absorbance values were measured. With the mass concentrations of iron in the iron standard series solutions as x-axis and the corresponding absorbance values as y-axis, plot the standard curve.

5.3.3 Determination of test sample

Under the same experimental conditions as those for the standard solution, the blank solution and the sample solution were introduced to the atomizer to determine the absorbance value, and the quantitative analysis was carried out with the standard series.

6. Expression of Analysis Result

The content of iron in the test sample is calculated according to Formula (1):

$$X = \frac{(\rho - \rho_0) \times V}{m} \qquad \cdots\cdots(1)$$

Where:

X ——the content of iron in the test sample, in mg/kg or mg/L;

ρ——the mass concentration of iron in the test sample solution used for determination, in mg/L;

ρ_0 ——the mass concentration of iron in the blank solution, in mg/L;

V ——the volume of the test sample digestion solution after being diluted to volume, in mL;

m ——the weighed amount or pipetted volume of the test sample, in g or mL.

When the content of iron is greater than or equal to 10.0 mg/kg or 10.0 mg/L, the calculation result shall remain

three significant figures; when the content of iron is less than 10.0 mg/kg or 10.0 mg/L, the calculation result shall remain two significant figures.

7. Precision

The absolute difference between the two independent determination results obtained under the repeatability conditions shall be no more than 10% of the mean arithmetical value.

8. Others

Calculated by weighing 0.5 g (or pipetting 0.5 mL) of sample and diluting to the volume of 25 mL, the limit of detection (LOD) and the limit of quantitation for this method is 0.75 mg/kg (or 0.75 mg/L) and 2.5 mg/kg (or 2.5 mg/L) respectively.

Method II Inductively Coupled Plasma Emission Spectrometry

Refer to GB 5009.268.

Method III Inductively Coupled Plasma Mass Spectrometry

Refer to GB 50069.268.

Annex A

Temperature programming of microwave digestion

See Table A.1 for the temperature programming of microwave digestion

Table A.1 Temperature programming of microwave digestion

Procedures	Pre-set temperature ℃	Temperature-rising time min	Holding time min
1	120	5	5
2	160	5	10
3	180	5	10

Annex B
Reference conditions of flame atomic absorption spectrometry

See Table B.1 for the reference conditions of flame atomic absorption spectrometry

Table B.1 Reference conditions of flame atomic absorption spectrometry

Element	Wavelength nm	Slit nm	Lamp current mA	Height of combustion head mm	Air flow rate L/min	Acetylene flow rate L/min
Iron	248.3	0.2	5-15	3	9	2