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# NATIONAL STANDARD OF THE

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GB 5009.90-2016

# National food safety standard -Determination of iron in foods

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# **Table of Contents**

Fc	preword	3
1	Scope	4
2	Principles	4
3	Reagents and materials	4
4	Instrument and equipment	5
5	Analytical procedures	6
6	Analysis results expression	8
7	Precision	9
8	Others	9
Αŗ	pendix A Microwave digestion temperature-rise program	.10
Δr	opendix B Reference conditions of flame atomic absorption spectrometry	11

GB 5009.90-2016

#### **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 (HNO<sub>3</sub>).
- **3.1.2** Perchloric acid (HClO<sub>4</sub>).
- 3.1.3 Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>).

#### 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.
- **3.2.2** Nitric acid solution (1+1): MEASURE 250 mL of nitric acid; POUR into 250 mL of water; MIX it uniformly.

GB 5009.90-2016

#### 5.2.2 Microwave digestion

Accurately WEIGH 0.2 g  $\sim$  0.8 g of solid sample (accurate to 0.001 g) or accurately MOVE 1.00 mL  $\sim$  3.00 mL of liquid sample into microwave digestion tank; ADD 5mL of nitric acid; DIGEST the sample according to the microwave digestion steps, digestion conditions refer to Table A.1; TAKE out the digestion tank after cooling; CATCH-acid to about 1.0 mL on a hot plate at 140°C  $\sim$  160°C. After cooling, TRANSFER the digestion juice into a 25 mL volumetric flask; USE a small amount of water to wash the inner tank and the inner caps for 2 $\sim$ 3 times; COMBINE washing liquid in the volumetric flask; Use water to dilute it to the mark; MIX it uniformly for spare-use. Meanwhile, DO a sample blank test.

#### 5.2.3 Pressure-tank digestion

Accurately WEIGH 0.3 g  $\sim$  2 g of solid sample (accurate to 0.001 g) or accurately MOVE 2.00 mL  $\sim$  5.00 mL of liquid sample into digestion inner-tank; ADD 5 mL of nitric acid; COVER inner-cap; TIGHTEN stainless steel outer-sheath; PLACE it into constant temperature oven at 140°C  $\sim$  160°C for 4h  $\sim$  5h. Slowly LOOSEN the outer tank after cooling; TAKE it out and DIGEST the inner tank; CATCH-acid to about 1.0 mL on adjustable electric hot plate at 140°C  $\sim$  160°C. After cooling, TRANSFER the digestion juice into a 25mL volumetric flask; USE a small amount of water to wash the inner tank and the inner caps for 2 $\sim$ 3 times; COMBINE washing liquid in the volumetric flask; Use water to dilute it to the mark; MIX it uniformly for spare-use. Meanwhile do a sample blank test.

#### 5.2.4 Dry digestion

Accurately WEIGH 0.5 g  $\sim$  3 g of solid sample (accurate to 0.001 g) or accurately MOVE 2.00 mL  $\sim$  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  $\sim$  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  $\sim$  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 $\sim$ 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 Determination

#### 5.3.1 Instrument's test conditions

Reference conditions are shown in Table B.1.

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