# NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

# GB 5009.17-2021

## Determination of total mercury and organic-mercury in foods

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**Issued by: National Health Commission;**
**State Administration for Market Regulation**

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## 1 Scope

Part 1 of this Standard specifies the methods for the determination of total mercury in food.

Part 1 of this Standard is applicable to the determination of total mercury in food.

Part 2 of this Standard specifies the methods for the determination of methyl mercury in food.

Part 2 of this Standard is applicable to the determination of methyl mercury in aquatic animals and their products, as well as rice and edible fungi.

Part 1.Determination of Total Mercury in Food Method 1 -- Atomic Fluorescence Spectrometry

## 2 Principle

After the specimen is heated and digested by acid, in the acidic medium, the mercury in the specimen is reduced to atomic-state mercury by potassium borohydride or sodium borohydride, which is brought into the atomizer by the carrier gas (argon).

Under the irradiation of mercury hollow cathode lamp, the ground-state mercury atoms are excited to a high-energy state. When they return from the high-energy state to the ground-state, they emit fluorescence with a characteristic wavelength. The fluorescence intensity is directly proportional to the mercury content, which is quantified by the external standard method.

## 3 Reagents and Materials

Unless it is otherwise specified, the reagents used in this Method are of top-grade purity, and the water is Grade-1 water specified in GB/T 6682.

### 3.2.1 Nitric acid solution (1 + 9). measure-take 50 mL of nitric acid; slowly add it to 450

mL of water; mix it up.

### 3.2.2 Nitric acid solution (5 + 95). measure-take 50 mL of nitric acid; slowly add it to

950 mL of water; mix it up.

3.2.3 Potassium hydroxide solution (5 g/L). weigh-take 5.0 g of potassium hydroxide;

use water to dissolve it and dilute to 1,000 mL; mix it up.

### 3.3 Standard Substance

Mercury chloride (HgCl2, CAS No.. 7487-94-7). purity >= 99%.

### 5.2.1 Method of microwave digestion

Weigh-take 0.2 g ~ 0.5 g of solid specimen (accurate to 0.001 g; for samples with relatively more water content, the sampling amount may be properly increased to 0.8 g), or accurately weigh-take 1.0 g ~ 3.0 g of liquid specimen (accurate to 0.001 g). For samples that are difficult to digest, for example, vegetable oils, weigh-take 0.2 g ~ 0.5 g (accurate to 0.001 g). Place it in a digestion tank; add 5 mL ~ 8 mL of nitric acid; put on the lid and let it stand for 1 h. For samples that are difficult to digest, add 0.5 mL ~

1 mL of hydrogen peroxide; tightly screw the lid; in accordance with the standard operating procedures of the microwave digestion apparatus (please refer to Table A.1 in Appendix A for the reference conditions of microwave digestion), conduct the digestion. After cooling it down, take it out; slowly open the lid of the tank to exhaust air;

### 5.2.2 Method of pressure tank digestion

Weigh-take 0.2 g ~ 1.0 g of solid specimen (accurate to 0.001 g; for samples with relatively more water content, the sampling amount may be properly increased to 2 g), or accurately weigh-take 1.0 g ~ 5.0 g of liquid specimen (accurate to 0.001 g). For samples that are difficult to digest, for example, vegetable oils, weigh-take 0.2 g ~ 0.5 g (accurate to 0.001 g). Place it in a digestion tank; add 5 mL of nitric acid; let it stand for 1 h or overnight. Put on the inner lid; tightly screw the stainless steel enclosure; put it in a constant-temperature drying oven; at 140 °C ~ 160 °C, maintain for 4 h ~ 5 h.

### 5.2.3.1 Grains

Weigh-take 1.0 g ~ 4.0 g (accurate to 0.001 g) of specimen; place it in a conical flask of the digestion device. Add several glass beads; add 45 mL of nitric acid and 10 mL and sulfuric acid; rotate the conical flask to prevent local carbonization. After installing the condenser pipe, heat it up at a low temperature. When it starts foaming, stop heating. After foaming stops, heat to reflux for 2 h. If the solution turns brown during the heating process, add 5 mL of nitric acid and continue to reflux for 2 h. Conduct digestion, until the sample is completely dissolved, which is generally pale yellow or colorless. After cooling, carefully add 20 mL of water from the upper end of the condenser pipe; continue to heat and reflux for 10 min. After leaving it to cool down, use an appropriate amount of water to rinse the

condenser pipe; incorporate the rinsing liquid into the digestive fluid. Filter the digestive fluid through glass wool into a 100 mL volumetric flask; use a small amount of water to wash the conical flask and filter.

Incorporate the washing liquid into the volumetric flask; add water to the scale; mix it up for later use. Meanwhile, conduct a blank test.

#### 5.2.3.5 Milk and dairy products

Weigh-take 1.0 g ~ 4.0 g (accurate to 0.001 g) of specimen, place it in a conical flask of the digestion device. Add several glass beads and 30 mL of nitric acid; for milk, add

10 mL of sulfuric acid; for dairy products, add 5 mL of sulfuric acid. Rotate the conical flask to prevent local carbonization. The subsequent steps are the same as 5.2.3.1 "After installing the condenser pipe, heat it up with a small fire... Meanwhile, conduct a blank test".

## 6 Expression of Analytical Results

The mercury content in the specimen shall be calculated in accordance with Formula(1).

## 7 Precision

When the mercury content in the sample is greater than 1 mg/kg, the absolute difference between two independent determination results obtained under repeatability conditions shall not exceed 10% of the arithmetic mean value. When the mercury content in the sample is less than or equal to 1 mg/kg, and greater than 0.1 mg/kg,

## 8 Others

When the mass of the sample is 0.5 g and the constant volume is 25 mL, the detection limit of this Method is 0.003 mg/kg;

## 9 Principle

After the sample is burned at a high temperature and is subject to catalytic pyrolysis, mercury is reduced to elemental mercury. Use gold amalgam for enrichment or directly bring into the detector by carrier gas. At a wavelength of 253.7 nm, measure the atomic absorption signal of mercury, or use a mercury lamp to excite the atomic fluorescence signal of the mercury being detected, which is quantified by the external standard method.

## 10 Reagents and Materials

Unless it is otherwise specified, the reagents used in this Method are of top-gradepurity,