



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

GB 5009.137-2025

Replacing GB 5009.137-2016

**National food safety standard - Determination of antimony in
foods**

食品安全国家标准 食品中锑的测定

Issued on: March 16, 2025

Implemented on: September 16, 2025

**Issued by: State Administration for Market Regulation;
Standardization Administration of the PRC**

Table of Contents

1 Scope
2 Principle
3 Reagents and Materials
3.1 Reagents
3.2 Preparation of Reagents
3.2.3 Thiourea-ascorbic acid solution. respectively weigh-take
3.2.4 Thiourea-potassium iodide solution. respectively weigh-take
3.2.5 Potassium hydroxide solution (
3.2.6 Potassium borohydride solution (20 g/L). weigh-take
3.2.10 Nitric acid solution (
3.4 Preparation of Standard Solutions
3.4.1 Antimony standard stock solution (1,000 mg/L). weigh-take
3.4.2 Antimony standard intermediate solution (100 mg/L). accurately pipette
3.4.3 Antimony standard working solution (
4 Instruments and Equipment
5 Analytical Steps...
5.2.2 Microwave digestion Weigh-take
5.2.3 Pressure tank digestion Weigh-take
6 Expression of Analytical Results...
7 Precision...

1 Scope

GB 5009.137-2025 is the Chinese national food safety method for antimony in food. Antimony reaches food from two directions: from the environment, in produce grown near mining and smelting areas, China being the world's largest antimony producer, and from packaging, since antimony trioxide is the catalyst used to make almost all PET, so a bottled drink held warm for long enough picks up a measurable amount. Both routes are regulated, and this is the method that supports the limits. The standard describes the determination by hydride generation atomic fluorescence spectrometry and by inductively coupled plasma mass spectrometry, covering for each the principle, the reagents and materials, the apparatus, the digestion of the sample, which for antimony is the step where the element is most easily lost, the preparation of the standard solutions, the analytical procedure, the calculation of results, the precision and the limit of quantification. The 2025 edition replaces

GB 5009.137-2016.

This Standard specifies the determination method of antimony in foods by hydride atomic fluorescence spectrometry and inductively coupled plasma mass spectrometry. This Standard is applicable to the determination of antimony in foods. Method I - Hydride Atomic Fluorescence Spectrometry

2 Principle

After the specimen is digested by acid heating, in an acidic medium, the antimony in the specimen reacts with sodium borohydride or potassium borohydride to generate volatile antimony hydride, which is carried into the atomizer by the carrier gas for atomization. Under the excitation of the antimony hollow cathode lamp, atomic fluorescence is generated, and its fluorescence intensity is proportional to the antimony content and is quantified by the external standard method.

3 Reagents and Materials

Unless otherwise specified, all reagents used in this Method are guaranteed reagents and the water is Grade-2 water specified in GB/T 6682.

3.1 Reagents

3.1.1 Nitric acid (HNO_3).

3.1.2 Hydrochloric acid (HCl).

3.1.3 Perchloric acid (HClO_4).

3.1.4 Thiourea $[(\text{NH}_2)_2\text{CS}]$. analytical grade.

3.1.5 Potassium iodide (KI). analytical grade.

3.1.6 Ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$). analytical grade.

3.1.7 Potassium borohydride (KBH_4) or sodium borohydride (NaBH_4).

3.1.8 Potassium hydroxide (KOH) or sodium hydroxide (NaOH).

3.1.9 Tartaric acid ($\text{C}_4\text{H}_6\text{O}_6$). purity 99.5%.

3.1.10 Argon (Ar). purity > 99.99%.

3.2 Preparation of Reagents

3.2.1 Nitric acid-perchloric acid mixture (10 + 1). measure-take 10 mL of perchloric acid and slowly add it into 100 mL of nitric acid and mix it well.

3.2.2 Hydrochloric acid solution (1 + 1). measure-take 50 mL of hydrochloric acid, add it to

50 mL of water and mix it well.

3.2.3 Thiourea-ascorbic acid solution. respectively weigh-take

10.0 g of thiourea and

10.0 g of ascorbic acid, dissolve in 100 mL of water, mix it well, and store it in a dark place. Prepare it immediately before use.

3.2.4 Thiourea-potassium iodide solution. respectively weigh-take

2.0 g of thiourea and

10.0 g of potassium iodide, dissolve in 100 mL of water, mix it well, and store it in a dark place. Prepare it immediately before use.

3.2.5 Potassium hydroxide solution (

5.0 g/L). weigh-take

5.0 g of potassium hydroxide, use water to dissolve it and dilute to 1,000 mL, and mix it well. Prepare it immediately before use. The potassium hydroxide in the solution may also be replaced by sodium hydroxide.

3.2.6 Potassium borohydride solution (20 g/L). weigh-take

10.0 g of potassium borohydride, use potassium hydroxide solution (

5.0 g/L) to dissolve it and dilute it to 500 mL, mix it well, and prepare it immediately before use. The potassium borohydride in the solution may also be replaced by

7.0 g of sodium borohydride.

3.2.7 Nitric acid solution (1 + 5). measure-take 100 mL of nitric acid, add it to 500 mL of water and mix it well.

3.2.8 Hydrochloric acid solution (5 + 95). measure-take 50 mL of hydrochloric acid, add it to 950 mL of water, and mix it well.

3.2.9 Nitric acid solution (1 + 1). measure-take 50 mL of nitric acid, add it to 50 mL of water, and mix it well.

3.2.10 Nitric acid solution (

2.5 mol/L). measure-take 173 mL of nitric acid, use water to dissolve it and dilute it to 1,000 mL, and mix it well.

3.3 Standard Substance Metallic antimony (Sb, CAS No.. 7440-36-0). purity > 99.99%.

3.4.1 Antimony standard stock solution (1,000 mg/L). weigh-take

0.50 g (accurate to 0.0001

g) of metallic antimony into a beaker, add

5.0 g of tartaric acid and

10.0 mL of nitric acid solution (1 + 1), after dissolution, transfer it to a 500 mL volumetric flask, use

2.5 mol/L nitric acid solution to reach a constant volume to the scale and mix it well. Place in a polyethylene plastic bottle, and store at room temperature away from light. The shelf life is 2 years. Or use antimony standard solution that has been certified by the state and awarded a standard substance certificate.

3.4.2 Antimony standard intermediate solution (100 mg/L). accurately pipette

1.00 mL of antimony standard stock solution (1,000 mg/L) into a 10 mL volumetric flask, add water to the scale and mix it well. Place it in a polyethylene plastic bottle, and store at 0 C ~ 5 C in a sealed container away from light. The shelf life is 6 months.

3.4.3 Antimony standard working solution (

1.00 mg/L). accurately pipette

1.00 mL of antimony standard intermediate solution (100 mg/L) into a 100 mL volumetric flask, add water to the scale and mix it well. Place it in a polyethylene plastic bottle, and store at 0 C ~ 5 C in a sealed container away from light. The shelf life is 1 month.

3.4.4 Antimony standard series solutions. respectively and accurately pipette 0 mL,

0.100 mL,

0.200 mL,

0.400 mL,

0.600 mL,

0.800 mL,

2.00 mL of antimony standard working solution (

1.00 mg/L) into 100 mL volumetric flasks, add a small amount of water to dilute them, then, add 10 mL of hydrochloric acid solution (1 + 1), 10 mL of thiourea-potassium iodide solution or thiourea-ascorbic acid solution, add water to reach a constant volume to the scale and mix them well. The mass concentration of the antimony standard series solutions is 0 g/L, 1.00 g/L, 2.00 g/L, 4.00 g/L, 6.00 g/L, 8.00 g/L, 10.0 g/L and 20.0 g/L. After leaving them for