NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 5009.33-2025

Replacing GB 5009.33-2016

National food safety standard - Determination of nitrite and nitrate in foods

食品安全国家标准 食品中亚硝酸盐与硝酸盐的测定

Issued on: September 2, 2025 **Implemented on: March 2, 2026**

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

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1 Scope

GB 5009.33 is the Chinese national food safety method for nitrite and nitrate in food. These two are the classic curing agents for meat, and they are also the classic contaminant of leafy vegetables and of drinking water; nitrite is acutely toxic at doses a child can reach, and the nitrosamines it forms are the long-term concern. The method matters in enforcement more than most: nitrite in cured meat and in pickled vegetables is one of the most frequently cited non-compliances in Chinese food inspection. The document gives the determination by ion chromatography and by spectrophotometry, and covers the principle, the reagents, the apparatus, the preparation of the sample, the analytical procedure, the calculation, the precision and the limit of quantification for each. The 2025 edition, effective 2 March 2026, replaces GB 5009.33-2016. It is a companion to the limits set in GB 2762 and in the product standards.

This Standard specifies the methods for determining nitrite and nitrate in foods. Method I of this Standard applies to the determination of nitrite and nitrate in foods (excluding foods for special medical purposes containing partially hydrolyzed milk protein, extensively hydrolyzed milk protein, amino acid formulas, and amino acid metabolism disorder formulas). Methods II and III of this Standard are applicable to the determination of nitrite and nitrate in foods. Method IV of this Standard is applicable to the determination of nitrate in fresh vegetables and fruits. Method I - Ion chromatography

2 Principle

After the sample is subject to protein precipitation and fat removal, extraction and purification, use potassium hydroxide or sodium hydroxide solution as eluent, separate by anion exchange column, and detect by conductivity detector or ultraviolet detector. Perform qualitative analysis using retention time, and quantitative analysis the external standard method.

3 Reagents and materials

Unless otherwise specified, all the reagents in this method are analytical reagents, and the water is grade-I water specified by GB/T 6682.

3.1 Reagents

3.1.1 Acetic acid (CH3COOH). chromatographic pure.

3.1.2 Potassium hydroxide (KOH).

3.2 Preparation of reagents

3.2.1 Acetic acid solution (3%). Measure 3 mL of acetic acid into a 100 mL volumetric flask; use water to dilute to the mark; mix well.

3.2.2 Potassium hydroxide solution (1 mol/L). Weigh

5.6 g of potassium hydroxide; dissolve it in freshly boiled and cooled water; dilute to 100 mL; mix well.

3.2.3 Sodium hydroxide solution (1 mol/L). Weigh

4.0 g of sodium hydroxide; add freshly boiled and cooled water to dissolve it; dilute to 100 mL; mix well.

3.3 Standards

3.3.1 Sodium nitrite standard (NaNO2, CAS No.. 7632-00-0). purity >= 99%. Or nitrite standards that have been certified by the state and granted a standard substance certificate.

3.3.2 Sodium nitrate standard (NaNO3, CAS No.. 7631-99-4). purity >= 99%. Or nitrate standards that have been certified by the state and granted a standard substance certificate.

3.4 Preparation of standard solution

3.4.1 Sodium nitrite standard stock solution (100 mg/L). Accurately weigh 0.100 0 g of sodium nitrite dried to constant weight at 110 °C~120 °C; use water to dissolve and dilute to 1 000 mL; mix well; store in a refrigerator at 2 °C ~ 6 °C. The validity period is 3 months.

3.4.2 Sodium nitrate standard stock solution (100 mg/L). Accurately weigh 0.100 0 g of sodium nitrate dried to constant weight at 110 °C ~ 120 °C; use water to dissolve and dilute to 1 000 mL; mix well; store in a refrigerator at 2 °C ~ 6 °C. The validity period is 3 months.

3.4.3 Mixed standard intermediate solution of sodium nitrite and sodium nitrate (

2.00 mg/L). Accurately transfer

2.00 mL of each of the standard stock solutions of sodium nitrite and sodium nitrate into 100 mL volumetric flasks; use water to dilute to the mark. Prepare when necessary.

3.4.4 Mixed standard solution of sodium nitrite and sodium nitrate. Transfer the intermediate solution of the mixed standard of sodium nitrite and sodium nitrate; add water to dilute it stepwise to prepare a series of standard solutions with mass concentrations of 0 mg/L, 0.010 0 mg/L, 0.020 0 mg/L, 0.050 0 mg/L,

0.100 mg/L,

0.200 mg/L,

0.500 mg/L,

2.00 mg/L. Prepare when necessary.

Note. The range of the standard curve can be adjusted according to the content of the target analyte in the sample, and it shall include at least 5 concentration levels (excluding zero).

3.5 Materials

3.5.1 Membrane needle filter. water-based, 0.22 μm.

3.5.2 C18 purification column. 1 mL, or equivalent column. Before use, activate with 10 mL of methanol and 15 mL of water in sequence.

3.5.3 Ag purification column. column capacity

2.0 ion equivalent ~

2.2 ion equivalent, or equivalent column. Use 10 mL of water to activate before use.

3.5.4 Na purification column. column capacity

2.0 ion equivalent ~