

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC

OF CHINA

中华人民共和国国家标准

GB 5009.33-2010

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

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Foreword

The Standard will replace the GB/T 5009.33-2008 "Determination of Nitrite and Nitrate in Foods".

Compared with the GB 5009.33-2008, main changes are following:

- In the Method I. the eluting condition is added for powder infant formula foods.
- The Method III: Oscillopolarography is deleted. and add determination of nitrite and nitrate in milk and dairy products as the Method III.

The former standards replaced by this standard are as following:

— GB 5009.33-85. GB/T 5009.33-1996. GB/T 5009.33-2003. GB/T 5009.33-2008.

National food safety standard Determination of nitrite and nitrate in foods

1. Scope

The Standard specifies a method of determining nitrite and nitrate in foods. The Standard is applicable to the determination of nitrite and nitrate in foods.

Method I Ion chromatography

2. Principle

Sample is extracted and purified using relevant method after protein precipitated and fat skimmed before separated by anion exchange column with KOH solution as an eluate and detected with a conductivity detector. It is then determined with an external standard method by taking retention time as for quantitative analysis.

3. Reagents and materials

3.1 Ultrapure water: with its conductivity of $18.2 \text{ M} \Omega$.cm.

- **3.2** CH₃COOH: analytically pure
- **3.3** KOH: analytically pure

3.4 CH3COOH solution (3%): 3ml CH3COOH (3.2) into 100ml volumetric flask. diluted to a mark with water and fully homogenized.

3.5 Nitrite ion (NO2⁻) stock solution (100mg/L. aqueous solution).

3.6 Nitrate ion (NO3⁻) stock solution (1000mg/L. aqueous solution).

3.7 Mixed standard solution of nitrate (counted on NO3- ion. the same herein below) and nitrite (counted on NO2⁻ ion. the same herein below): accurately pipette 1.0mLof nitrite ion (NO2⁻) stock solution and nitrate ion (NO3⁻) stock solution to 100mL volumetric flask, diluted to a mark with water. which 1mL of this solution contains 1.0mg of nitrite ion and 10.0mg of nitrate ion.

4. Instruments and equipments

4.1 Ion chromatograph: including a conductivity detector suppressor high capacity anion exchange column. measuring ring in $25 \ \mu$ L.

4.2 Food disintegrator.

4.3 Supersonic cleaner.

4.4 Analytical balance: readability 0.1mg and 1mg.

4.5 Centrifuge: rotational speed no less than 10000rpm with 5ml or 10ml centrifugal tubes.

4.6 $0.22 \,\mu$ m syringe filters with hydrophilic filterable membrane.

4.7 Decontaminating column: including C₁₈ column, Ag column and Na column or its equivalent.

4.8 Syringe: 1.0ml and 2.5ml.

NOTE: All glassware should be soaked in 2mol/L of NaOH solution and water for 4h, respectively, followed by rinsing with water for 3-5 times before ready for use later.

5. Analytical Procedures

5.1 Sample pre-treatment

5.1.1 Fresh vegetable and fruit: the whole piece of vegetable and fruit is washed with deionized water, the edible portion of these vegetable and fruit is then disintegrated to uniformity after air dried. The adequate amount of disintegrated sample is then taken by quartering, and prepared into slurry with a stamp mill for use later. Water addition should be recorded if water is required to add.

5.1.2 Meat, egg, aquatic products and their processed products: an adequate amount or full of materials is taken with quartering, and then prepared into slurry with a stamp mill for use later.

5.1.3 Solid dairy products such as milk powder, soybean milk powder, and infant formula powder (excluding cheese): sample is put in a container with lid in capacity of two folds of sample; the sample is finally homogenized by repeatedly shaking and reversing the container.

5.1.4 Fermented milk, milk, condensed milk and other liquid dairy products: the sample isc or repeatedly shaking and reversing the container.

5.1.5 Cheese: an adequate amount of sample is uniformly ground to a muddy form. In order to avoid the loss of water content the excessive heat should be avoidable during the process of grinding.

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