NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 5009.26-2023

National food safety standard - Determination of N-nitrosamines in foods

食品安全国家标准 食品中N-亚硝胺类化合物的测定

Issued on: September 6, 2023 **Implemented on: March 6, 2024**

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

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Foreword

This document was issued on 6 September 2023 by the State Administration for Market Regulation; Standardization Administration of the PRC and takes effect on 6 March 2024.

It is a GB standard without the /T suffix: compliance is mandatory in China.

1 Scope

GB 5009.26-2023 covers the laboratory determination of N-nitrosamines in food, and lays out four independent analytical routes for the same analyte, N-dimethylnitrosamine, so a laboratory can use the instrument it owns. Each route is built the same way: principle, reagents and materials, instruments and apparatuses, analysis steps, expression of results, precision, and a closing clause on detection and quantitation limits. The first and third routes carry the sample through steam distillation, absorb the nitrosamine into dichloromethane, separate it by liquid-liquid extraction and measure it by gas chromatography with tandem mass spectrometry; the second drops distillation for acetonitrile extraction with a dispersive filler cleaning the matrix; the fourth ends at a thermal energy analyser, the detector still in service where no mass spectrometer is available. Three of the four add a deuterated internal standard before work-up, which keeps recovery honest when a fatty meat matrix holds back analyte during distillation. Nitrosamines form in cured and smoked products from nitrite and amines and matter at concentrations no taste panel would notice, so a shipment is accepted or rejected on a number two laboratories have to agree on - hence the repeatability clause and the appendix drawing the steam distillation device. Written for food testing laboratories, meat product processors, and the regulators who arbitrate a failed result.

This standard specifies the method for the determination of N-dimethylnitrosamine (NDMA) in foods.

This standard applies to the determination of N-dimethylnitrosamine in meat and meat products, aquatic animals and their products.

Method 1.Steam distillation-gas chromatography-mass spectrometry/mass spectrometry

2 Principle

This method uses N-dimethylnitrosamine-D6 as the internal standard. The internal standard is added to the sample; after steam distillation, the N-dimethylnitrosamine in the sample is absorbed by dichloromethane and separated by liquid-liquid extraction,

which is determined by gas chromatography-mass spectrometry/mass spectrometry (GC-MS/MS) and quantified by the internal standard method.

3 Reagents and materials

Unless otherwise specified, all the reagents in this method are analytical reagents, and the water is grade-1 water specified by GB/T 6682.

3.1 Reagents

3.1.1 Dichloromethane (CH2Cl2). chromatographic pure.

3.1.2 Concentrated sulfuric acid (H2SO4). 18.4 mol/L.

3.1.3 Isooctane (C8H18). chromatographic pure.

3.1.4 Anhydrous sodium sulfate (Na2SO4).

3.1.5 Sodium chloride (NaCl). guaranteed reagent.

3.2 Preparation of reagents

Sulfuric acid solution (1+3). Measure 30 mL of concentrated sulfuric acid; slowly pour it into 90 mL of cold water; stir to fully dissipate heat; mix carefully after cooling.

3.3 Standards

3.3.1 N-dimethylnitrosamine standard solution (C2H6N2O, CAS number. 62-75-9). N-dimethylnitrosamine methanol solution with a mass concentration of 1 000 ug/mL, or standard products certified by the nation and awarded a standard material certificate.

3.3.2 N-dimethylnitrosamine-D6 internal standard solution (NDMA-D6, C2D6N2O, CAS number. 17829-05-9). the mass concentration is 1 000 ug/mL, and the solvent is methanol.

3.4 Preparation of standard solutions

3.4.1 N-dimethylnitrosamine standard stock solution (100 ug/mL). Accurately draw 1.0 mL of N-dimethylnitrosamine standard solution (1 000 ug/mL); put it in a 10 mL volumetric flask; use dichloromethane to adjust the volume to the mark; mix well. Transfer the solution to a brown glass container; store at -18 °C in the dark for 6 months.

3.4.2 N-dimethylnitrosamine standard intermediate solution (1 ug/mL). Accurately draw 1.0 mL of N-dimethylnitrosamine standard stock solution (100 ug/mL); place it

in a 100 mL volumetric flask; use dichloromethane to adjust the volume to the mark; mix well. Transfer the solution to a brown glass container; store at -18 °C in the dark for 3 months.

3.4.3 N-dimethylnitrosamine-D6 internal standard stock solution (100 ug/mL). Accurately draw 1.0 mL of N-dimethylnitrosamine-D6 standard solution; place it in a 10 mL volumetric flask; use dichloromethane to adjust the volume to the mark; mix well. Transfer the solution to a brown glass container; store at -18 °C in the dark for 6 months.

3.4.4 N-dimethylnitrosamine-D6 internal standard intermediate solution (1 ug/mL). Accurately draw 1.0 mL of N-dimethylnitrosamine-D6 internal standard stock solution; place it in a 100 mL volumetric flask; use dichloromethane to adjust the volume to the mark; mix well. Transfer the solution to a brown glass container; store at -18 °C in the dark for 3 months.

3.4.5 N-dimethylnitrosamine standard and internal standard mixed series working solutions. Respectively and accurately draw 0.1 mL, 0.2 mL, 0.5 mL, 1.0 mL and 2.0 mL of N-dimethylnitrosamine standard intermediate solution (1 ug/mL); place them in the 10 mL volumetric flasks; add 0.4 mL of internal standard intermediate solution (1 ug/mL) to each; use dichloromethane to adjust the volume to the mark; mix well. The mass concentrations of N-dimethylnitrosamine standard series working solutions are 10 ug/L, 20 ug/L, 50 ug/L, 100 ug/L and 200 ug/L, among which the mass concentrations of the internal standards are all 40 ug/L. Prepare when necessary.

4 Instruments and apparatuses

4.1 Gas chromatography-mass spectrometry/mass spectrometry (GC-MS/MS).

4.2 Rotary evaporator.

4.3 All-glass steam distillation device or fully automatic steam distillation device (see Figure A.1 in Appendix A).

4.4 Nitrogen-blowing instrument.

4.5 Electric balance. sensitivity of 0.001 g.