NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 5009.97-2023

National food safety standard - Determination of sodium cyclamate in foods

食品安全国家标准 食品中环己基氨基磺酸盐的测定

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 5009.97-2023 measures sodium cyclamate in food. Three methods share the document, each with its own principle, reagents and materials, instruments and equipment, analysis steps, expression of analysis results, precision and closing clause. The first is gas chromatography: the cyclamate is extracted from the sample with water, reacted with sodium nitrite in a sulfuric acid medium to form cyclohexanol nitrite and cyclohexanol, taken up in n-heptane, and measured on a gas chromatograph with a hydrogen flame ionisation detector against the chromatogram of the derivatives in Annex A. The second passes the derivatised extract through a liquid chromatograph fitted with an ultraviolet or diode array detector, with the reference chromatogram in Annex B. The third extracts the sample and measures it by liquid chromatography-tandem mass spectrometry, identifying the analyte by retention time and relative ion abundance, with the multiple reaction monitoring chromatogram in Annex C. Cyclamate is a permitted sweetener whose use limit varies with the food category, and at the levels in dispute it leaves no clue in taste or appearance, so a compliance argument can only be settled by analysis - which is why one agreed extraction and one agreed set of confirmation criteria matter more than the choice of instrument. For food inspection laboratories, beverage and confectionery producers, and market regulators.

This Standard specifies the method for the determination of cyclohexyl sulfamate in food.

Method One -- Gas Chromatography is applicable to the determination of cyclohexyl sulfamate in food (except distilled wine, fermented wine, prepared wine, cooking wine and other ethanol-containing foods).

Method Two -- Liquid Chromatography is applicable to the determination of cyclamate in food.

Method Three -- Liquid Chromatography-Mass Spectrometry/Mass Spectrometry is applicable to the determination of cyclamate in food.

Method One -- Gas Chromatography

2 Principle

The cyclamate in the specimen is extracted with water. It reacts with sodium nitrite in sulfuric acid medium to generate cyclohexanol nitrite and cyclohexanol. After extraction with n-heptane, use gas chromatography-hydrogen flame ionization detector to determine. Use external standard method to quantify.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method are analytically pure, and the water is grade one water specified in GB/T 6682.

3.1 Reagents

3.1.1 n-Heptane [CH3(CH2)5CH3]. chromatographically pure.

3.1.2 Petroleum ether. Boiling range is 30 degrees C~60 degrees C.

3.1.3 Sulfuric acid (H2SO4). 95.0%~98.0% (mass fraction); density. 1.84 g/mL.

3.1.4 Sodium nitrite (NaNO2).

3.1.5 Amylase. enzyme activity >=1500 U/g.

3.1.6 Absolute ethanol (CH3CH2OH).

3.2 Preparation of reagents

3.2.1 Sodium nitrite solution (50 g/L). Weigh 50 g of sodium nitrite, dissolve in water and dilute to 1000 mL. Mix well.

3.2.2 Sulfuric acid solution (200 g/L). Measure 108 mL of sulfuric acid and slowly add it to 800 mL of water. Stir constantly to avoid localized overheating. After cooling, add water to dilute to 1000 mL. Mix well.

3.3 Standard product

Sodium cyclohexyl sulfamate standard (C6H12NSO3Na). purity >=99%, or a standard certified by the country and awarded a reference material certificate.

3.4 Preparation of standard solution

3.4.1 Cyclohexylsulfamic acid standard stock solution (6 mg/mL). Accurately weigh an appropriate amount of sodium cyclamate standard (accurate to 0.1 mg) into a 100 mL volumetric flask. Dissolve in water and bring the volume to the mark. Mix well (the conversion factor of sodium cyclohexylsulfamate into cyclohexylsulfamate is 0.8907). Transfer the solution to a brown glass container. Store at 4°C away from light. It is valid for 12 months.

3.4.2 Cyclohexylsulfamic acid standard intermediate solution (1200 ug/mL). Accurately pipette 10 mL of cyclamate standard stock solution into a 50 mL volumetric

flask. Use water to bring the volume to the mark. Mix well. Transfer the solution to a brown glass container. Store at 4°C away from light. It is valid for 6 months.

3.5 Materials

3.5.1 Centrifuge tube. 50 mL graduated centrifuge tube with screw cap.

3.5.2 Organic phase microporous filter membrane. 0.45 um in pore size.

4 Instruments and equipment

4.1 Gas chromatograph. equipped with hydrogen flame ionization detector (FID).

4.2 Analytical balance. division is 1 mg, 0.1 mg.

4.3 Vortex mixer.

Vortex for 5 min. Perform ultrasonic extraction for 30 min. Mix well and bring to room temperature.

5.2.2 Specimens of jelly, candy, rice flour, starch products, etc.

Weigh 2 g of specimen (accurate to 0.001 g) into a centrifuge tube. Add 20 mL of water (0.2 g of amylase is required for specimens such as rice flour and starch products). After mixing evenly, heat in a water bath at 60 degrees C+/-2 degrees C for 20 min. Vortex for 5 min. Perform

ultrasonic extraction for 10 min. Mix well and bring to room temperature.

5.2.3 Other solid and semi-solid specimens

Weigh 2 g of specimen (accurate to 0.001 g) into a centrifuge tube. Add 20 mL of water. Vortex for 5 min. Perform ultrasonic extraction for 30 min. Mix well and bring to room temperature.

5.2.4 Liquid specimen

Weigh 2 g of specimen (accurate to 0.001 g) into a centrifuge tube. Add water to 20 mL. Vortex for 5 min. Perform ultrasonic extraction for 10 min. Mix well and bring to room temperature.

5.3 Derivatization

Place the centrifuge tube containing the specimen extract solution in an ice bath for 10 min, then add 10 mL of n-heptane, 5 mL of sodium nitrite solution (50 g/L), and 5 mL