



GB 5009.97-2023

National Food Safety Standard

Determination of Cyclohexylaminosulfonate in Food

食品安全国家标准 食品中环己基氨基磺酸盐的测定

National Health Commission of the People's
Republic of China
State Administration for Market Regulation

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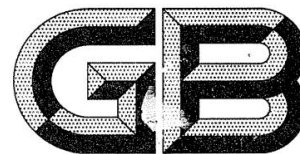
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National Standard of the People's Republic of China

GB 5009.97-2023

In Replacement of GB 5009.97-2016

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Determination of Cyclohexylaminosulfonate in Food

食品安全国家标准

食品中环己基氨基磺酸盐的测定

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Foreword

This standard replaces GB 5009.97-2016 "National Food Safety Standard - Determination of Sodium Cyclamate in Foods".

Compared with GB 5009.97-2016, the main changes of this standard are as follows:

——Change the name of the standard to "National Food Safety Standard - Determination of Cyclohexylaminosulfonate in Food";

——The scope of application has been modified;

——The pre-processing method was modified;

——The instrument conditions and parameters were modified;

——The quantification method of the third method, liquid chromatography-mass spectrometry/mass spectrometry, was modified to the internal standard method.

National Food Safety Standard

Determination of Cyclohexylaminosulfonate in Food

1 Scope

This standard specifies the determination method of Cyclohexylaminosulfonate in food.

The first method, gas chromatography, is applicable to the determination of cyclohexylaminosulfonate in foods (except distilled wine, fermented wine, blended wine, cooking wine and other foods containing ethanol).

The second method, liquid chromatography, is suitable for the determination of cyclohexylaminosulfonate in food.

The third method, liquid chromatography-mass spectrometry/mass spectrometry, is suitable for the determination of cyclohexylaminosulfonate in food.

Method 1 Gas Chromatography

2 Principle

The cyclohexylaminosulfonate in the sample is extracted with water, reacted with sodium nitrite in a sulfuric acid medium to generate cyclohexanol nitrite and cyclohexanol, extracted with n-heptane, and then determined by gas chromatography-hydrogen flame ionization detector and quantified by external standard method.

3 Reagents and materials

Unless otherwise specified, all reagents used in this method were of analytical grade and the water was Grade-1 water as specified in GB/T6682.

3.1 Reagents

3.1.1 n-Heptane [$\text{CH}_3(\text{CH}_2)_5\text{CH}_3$]: chromatographically pure.

3.1.2 Petroleum ether: boiling range is $30^\circ\text{C}\sim 60^\circ\text{C}$.

3.1.3 Sulfuric acid (H_2SO_4), 95.0%~98.0% (mass fraction), density: 1.84g/mL.

3.1.4 Sodium nitrite (NaNO_2).

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3.1.5 Amylase: enzyme activity $\geq 1500\text{U/g}$.

3.1.6 Anhydrous ethanol ($\text{CH}_3\text{CH}_2\text{OH}$).

3.2 Reagent preparation

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

3.2.2 Sulfuric acid solution (200g/L): Measure 108mL of sulfuric acid and slowly add it into 800mL of water, stirring constantly to avoid local overheating. After cooling, dilute with water to 1000mL and mix well.

3.3 Standards

Sodium cyclohexylaminosulfonate standard ($\text{C}_6\text{H}_{12}\text{NSO}_3\text{Na}$): purity $\geq 99\%$, or standard products certified by the state and awarded with standard material certificate.

3.4 Preparation of standard solution

3.4.1 Cyclamic 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 it in water and dilute to the mark, and mix well (the conversion factor of sodium cyclamate to cyclamic acid is 0.8907). Transfer the solution to a brown glass container and store it at 4°C away from light. The shelf life is 12 months.

3.4.2 Cyclamic acid standard intermediate solution (1200 $\mu\text{g/mL}$): Accurately pipette 10 mL of cyclamic acid standard stock solution into a 50 mL volumetric flask, dilute to the mark with water, and mix well. Transfer the solution to a brown glass container and store it at 4°C away from light. The shelf life is 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 μm pore size.

4. Instruments and equipment

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



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