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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 31604.56-2023

National food safety standard - Food contact materials and products - Determination of laurolactam migration

食品安全国家标准 食品接触材料及制品 月桂内酰胺迁移量的测定

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 31604.56-2023 is the national food safety method for measuring how much laurolactam migrates out of food contact materials and articles. Two methods run in parallel through the document. The first works by liquid chromatography: once the migration test is done, aqueous, acidic and ethanol-containing food simulants, together with the ethanol solvent used in place of fatty food, are injected directly. The second works by liquid chromatography with tandem mass spectrometry on the same simulants. Each method carries its own principle, reagents and materials, instruments and equipment, analysis steps, expression of results, precision, and a clause of further provisions; Annex A shows the chromatogram of the laurolactam standard solution, so an analyst can confirm which peak is being

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integrated. Laurolactam is the monomer from which polyamide 12 is built, and whatever is left unreacted in the polymer moves into what the article holds - faster as the food gets warmer, fattier and stays in contact longer. Without one prescribed procedure, two laboratories testing the same tubing or film can report figures that differ by more than the limit they are being judged against, and enforcement becomes a matter of which laboratory was used. Mandatory. For makers and importers of food contact plastics and for the testing and market supervision laboratories that check them.

This Standard specifies the method for determining the migration amount of laurolactam in food contact materials and products.

This Standard is applicable to the determination of the migration amount of laurolactam in polyamide food contact materials and products.

Method one -- Liquid chromatography

2 Principle

After the migration test of food contact materials and products, liquid chromatography is used for testing. Among them, water-based, acidic, ethanol-containing food simulants and chemical alternative solvents of 95% (volume fraction) ethanol are directly injected after filtration. Oil-containing food simulants are filtered and injected after liquid-liquid extraction and solid-phase extraction. The chemical alternative solvent isooctane is injected by filtration after solvent removal and reconstitution with acetonitrile. Use retention time to qualitative determination. Use external standard method for quantification.

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 Acidic, ethanol-containing, grease-containing food simulants and chemical alternative solvents: The reagents used are in accordance with the regulations of GB 5009.156.

3.1.2 Acetonitrile (C2H3N): chromatographically pure.

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3.1.3 Formic acid (CH2O2): chromatographically pure.

3.1.4 Ammonia water: the mass fraction of ammonia is 25%~28%.

3.1.5 Absolute ethanol (C2H6O).

3.2 Reagent preparation

3.2.1 Formic acid-water solution: measure 5.0 mL of formic acid and 95 mL of water, and mix well.

3.2.2 Ammonia-acetonitrile solution: measure 2.0 mL of ammonia, 8.0 mL of water and 90 mL of acetonitrile, and mix well.

3.2.3 Formic acid-acetonitrile-water solution: measure 20 mL of acetonitrile and 80 mL of water; pipette 100 uL of formic acid; mix well.

3.3 Standard product

Laurolactam standard product (C12H23NO, CAS: 947-04-6, also known as laurolactam or azacyclotridecane-2-one): purity >=98%, or certified by the country and awarded as a standard material certificate of reference material.

3.4 Standard solution preparation

3.4.1 Laurolactam standard stock solution (1000 mg/L): Weigh 100 mg (accurate to 0.1 mg) of laurolactam standard product. Dissolve in absolute ethanol and adjust the volume to 100 mL. Shake well. Transfer the solution to a brown glass container. Store at 4°C away from light. Validity period is 3 months.

3.4.2 Laurolactam standard intermediate solution (200 mg/L): Pipette 5.0 mL of laurolactam standard stock solution into a 25 mL volumetric flask. Dilute to volume with absolute ethanol. Shake well. Transfer the solution to a brown glass container. Store at 4°C away from light. Validity period is 3 months.

3.4.3 Aqueous, acidic, ethanol-containing food simulants or 95% (volume fraction) ethanol standard working solution: Pipette 0.50 mL, 1.0 mL, 2.5 mL, 5.0 mL, and 10 mL of laurolactam standard intermediate solution into 100 mL volumetric flasks. Dilute to volume with corresponding aqueous, acidic, ethanol-containing food simulant or 95% (volume fraction) ethanol. Prepare standard working solutions with concentrations of

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1.0 mg/L, 2.0 mg/L, 5.0 mg/L, 10 mg/L and 20 mg/L, respectively. Prepare when needed.

3.4.4 Standard working solution of oil-containing food simulants: Weigh 2 g (accurate to 0.01 g) of olive oil. Add 20 uL, 30 uL, and 50 uL of laurolactam standard intermediate solution and 20 uL and 40 uL of laurolactam standard stock solution, respectively. Mix well. Prepare standard working solutions with concentrations of 2.0 mg/kg, 3.0 mg/kg, 5.0 mg/kg, 10 mg/kg and 20 mg/kg. Prepare when needed. Before putting on the machine, follow 5.1.3 to process it simultaneously with the soaking solution of oil-containing food simulants.

5.1.3 Fat-containing food simulants test solution

Weigh 2 g (accurate to 0.01 g) of the oil-containing food simulant soaking solution into a 15 mL centrifuge tube. Add 4.0 mL of acetonitrile. Vortex and extract for 10 min. Centrifuge at 8500r/min for 2 min. Take the supernatant. Repeat extraction of soaking liquid once. Combine the two extracts. Concentrate to approximately 0.5 mL with nitrogen blowing at 45°C. After reconstitution with 4.0 mL of formic acid-aqueous solution, transfer to a mixed cation solid-phase extraction cartridge activated with 3.0 mL of acetonitrile, pure water, and formic acid-aqueous solution. Elute with 3.0 mL of formic acid-water solution and acetonitrile in sequence. Elute with 3.0 mL of ammonia-acetonitrile solution. The eluate is blown dry with nitrogen at 45°C. Add 1.0 mL of formic acid-acetonitrile-water solution. Vortex for 1 min to reconstitute. Filter with microporous membrane for measurement.

5.1.4 Isooctane test solution

Pipette 1.0 mL of the isooctane soaking solution obtained from the migration test. Blow dry with nitrogen at 45°C. Add 1.0 mL of acetonitrile to dissolve the residue. Filter with microporous membrane for measurement.

5.1.5 Preparation of blank test solution

Food simulants and chemical alternative solvents that are not in contact with food contact materials and products are processed according to 5.1.2, 5.1.3 and 5.1.4 to

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