

Translated English of Chinese Standard: GB31604.56-2023

www.ChineseStandard.net → Buy True-PDF → Auto-delivery.

Sales@ChineseStandard.net

GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 31604.56-2023

**National Food Safety Standards -- Food Contact Materials
and Products -- Determination of Migration Amount of
Lauro lactam**

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 06, 2023

Implemented on: March 06, 2024

**Issued by: National Health Commission of the People's Republic of China;
State Administration for Market Regulation.**

Table of Contents

1 Scope	3
2 Principle	3
3 Reagents and materials	3
4 Instruments and equipment	5
5 Analysis steps.....	5
6 Expression of analysis results.....	7
7 Precision.....	9
8 Other	9
9 Principle	10
10 Reagents and materials	10
11 Instruments and equipment.....	11
12 Analysis steps	12
13 Expression of analysis results	14
14 Precision	14
15 Other	14
Annex A Chromatogram of lauro lactam standard solution.....	16

National Food Safety Standards -- Food Contact Materials and Products -- Determination of Migration Amount of Lauro lactam

1 Scope

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

This Standard is applicable to the determination of the migration amount of lauro lactam 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 (C_2H_3N): chromatographically pure.

3.1.3 Formic acid (CH_2O_2): 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 µL of formic acid; mix well.

3.3 Standard product

Lauro lactam standard product (C₁₂H₂₃NO, CAS: 947-04-6, also known as lauro lactam 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 Lauro lactam standard stock solution (1000 mg/L): Weigh 100 mg (accurate to 0.1 mg) of lauro lactam 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 Lauro lactam standard intermediate solution (200 mg/L): Pipette 5.0 mL of lauro lactam 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 lauro lactam 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 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 µL, 30 µL, and 50 µL of lauro lactam standard intermediate solution and 20 µL and 40 µL of lauro lactam 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 obtain a blank test solution.

5.2 Instrument reference conditions

The instrument reference conditions are as follows:

- a) Chromatographic column: C₁₈ column; column length is 250 mm; column inner diameter is 4.6 mm; particle size is 5 µm, or a chromatographic column with equivalent performance;
- b) Mobile phase: acetonitrile and water (50+50, volume ratio);
- c) Flow rate: 1.0 mL/min;
- d) Injection volume: 20 µL;
- e) Column temperature: 25°C;
- f) Detection wavelength: 210 nm.

5.3 Preparation of standard curve

Determine the standard working solution according to the instrument reference conditions listed in 5.2. Draw a standard curve with the concentration of lauro lactam in

the standard working solution as the abscissa and the corresponding peak area as the ordinate. The reference chromatogram of lauro lactam standard solution is shown in Figure A.1 of Annex A.

5.4 Determination of test solution

Measure the food simulant test solution and blank test solution according to the instrument reference conditions listed in 5.2. Use the retention time to determine the peak area of the target substance. Obtain the concentration of lauro lactam in the test solution according to the standard curve. Calculate the migration amount of lauro lactam according to Chapter 6. The deviation between the retention time of the chromatographic peak of the food simulant test solution and the retention time of the chromatographic peak of the standard working solution shall be within the range of $\pm 2.5\%$.

The test solution can be diluted according to specific circumstances so that the measured value is within the linear range of the standard curve.

6 Expression of analysis results

6.1 Calculation of migration amount of lauro lactam in food simulants

The migration amount of lauro lactam in food simulants is calculated according to formula (1).

$$X_1 = (c - c_0) \cdot N \quad \dots\dots\dots (1)$$

Where,

X_1 - The migration amount of lauro lactam in food simulants, in milligrams per liter (mg/L) or milligrams per kilogram (mg/kg);

c - The content of lauro lactam in the food simulant test solution, in milligrams per liter (mg/L) or milligrams per kilogram (mg/kg);

c_0 - The content of lauro lactam in the blank test solution, in milligrams per liter (mg/L) or milligrams per kilogram (mg/kg);

N - The dilution factor.

6.2 Calculation of specific migration amounts of lauro lactam in food contact materials and products

6.2.1 Calculation of specific migration amount of lauro lactam in non-sealed food contact materials and products (expressed in mg/kg)

simulants and chemical alternative solvents 95% (volume fraction) ethanol and isooctane is 0.5 mg/L. The limit of quantitation is 1.0 mg/L. The method detection limit of lauro lactam in oil-containing simulants is 1.0 mg/kg. The limit of quantification is 2.0 mg/kg.

The detection limit and quantitation limit of the specific migration amount of lauro lactam in food contact materials and products are based on the method detection limit and quantification limit of lauro lactam in food simulants. Conversion is performed according to Chapter 6.

Method two -- Liquid chromatography-tandem mass spectrometry

9 Principle

After the migration test of food contact materials and products, liquid chromatography-tandem mass spectrometry is used for detection. 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. Retention time and relative ion abundance ratio are used for qualitative determination. Use external standard method for quantification.

10 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.

10.1 Reagents

Same as 3.1.

10.2 Solution preparation

Same as 3.2.

10.3 Standard product

Same as 3.3.

10.4 Standard solution preparation

10.4.1 Laurolactam standard stock solution (1000 mg/L): same as 3.4.1.

10.4.2 Laurolactam standard intermediate solution (10 mg/L): Pipette 1 mL of laurolactam standard stock solution into a 100 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. It is valid for 3 months.

10.4.3 Laurolactam standard intermediate solution (1.0 mg/L): Pipette 1 mL of laurolactam standard stock solution into a 10 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. It is valid for 3 months.

10.4.4 Aqueous, acidic, ethanol-containing food simulants and 95% (volume fraction) ethanol standard working solution: Accurately pipette 0.30 mL, 0.50 mL, 1.0 mL, 5.0 mL and 10 mL of laurolactam standard intermediate solution (1.0 mg/L) into 100 mL volumetric flasks. Dilute to volume with corresponding aqueous, acidic, ethanol-containing food simulants or 95% (volume fraction) ethanol. Prepare 0.003 mg/L, 0.005 mg/L, 0.010 mg/L, 0.050 mg/L, and 0.10 mg/L standard working solutions. Prepare when needed.

10.4.5 Standard working solution of oil-containing food simulants: Pipette 12 µL, 20 µL, and 40 µL of laurolactam standard intermediate solution (1.0 mg/L) and 10 µL and 20 µL of laurolactam standard intermediate solution (10 mg/L) respectively. Add to 2 g (accurate to 0.01 g) of oil-containing food simulant. Mix well. Prepare 0.006 mg/kg, 0.010 mg/kg, 0.020 mg/kg, 0.050 mg/kg, and 0.10 mg/kg standard working solutions. Prepare when needed. Before putting on the machine, follow 12.1 to synchronize with the oil-containing food simulant soaking solution.

10.4.6 Iso-octane standard working solution: Pipette 0.30 mL, 0.50 mL, 1.0 mL, 5.0 mL and 10 mL of laurolactam standard intermediate solution (1.0 mg/L) into 100 mL volumetric flasks, respectively. Dilute to volume with isooctane. The concentrations of the standard working solutions are 0.003 mg/L, 0.005 mg/L, 0.010 mg/L, 0.050 mg/L, and 0.10 mg/L, respectively. Prepare when needed. Before putting on the machine, process it simultaneously with the isooctane soaking solution according to 12.1.

11 Instruments and equipment

11.1 Liquid chromatography-tandem mass spectrometer: equipped with electrospray ion source (ESI).

11.2 Adjustable pipette: the measuring range is 10 µL~100 µL and the measuring range is 100 µL~1000 µL.

11.3 Analytical balance: division is 0.0001 g (0.1 mg) and 0.01 g.

11.4 Organic microporous filter membrane: 0.45 µm.

11.5 Vortex mixer.

11.6 Centrifuge.

11.7 Nitrogen concentration device.

11.8 Solid phase extraction device.

11.9 Mixed cationic solid-phase extraction cartridge or equivalent purification column:
150 mg/6 mL.

12 Analysis steps

12.1 Sample preparation

Same as 5.1.

12.2 Instrument reference conditions

12.2.1 Liquid chromatography conditions

The reference conditions for liquid chromatography are as follows:

- a) Chromatographic column: C₁₈ chromatographic column; column length is 100 mm; column inner diameter is 2.1 mm; particle size is 1.7 μm, or a column with equivalent performance;
- b) Mobile phase: A is an aqueous solution containing 0.1% (volume fraction) formic acid; B is an acetonitrile solution containing 0.1% (volume fraction) formic acid; the elution gradient is shown in Table 1;
- c) Flow rate: 0.3 mL/min;
- d) Column temperature: 40°C;
- e) Injection volume: 2 μL.

This is an excerpt of the PDF (Some pages are marked off intentionally)

Full-copy PDF can be purchased from 1 of 2 websites:

1. <https://www.ChineseStandard.us>

- SEARCH the standard ID, such as GB 4943.1-2022.
- Select your country (currency), for example: USA (USD); Germany (Euro).
- Full-copy of PDF (text-editable, true-PDF) can be downloaded in 9 seconds.
- Tax invoice can be downloaded in 9 seconds.
- Receiving emails in 9 seconds (with download links).

2. <https://www.ChineseStandard.net>

- SEARCH the standard ID, such as GB 4943.1-2022.
- Add to cart. Only accept USD (other currencies - <https://www.ChineseStandard.us>).
- Full-copy of PDF (text-editable, true-PDF) can be downloaded in 9 seconds.
- Receiving emails in 9 seconds (with PDFs attached, invoice and download links).

Translated by: Field Test Asia Pte. Ltd. (Incorporated & taxed in Singapore. Tax ID: 201302277C)

About Us (Goodwill, Policies, Fair Trading...): <https://www.chinesestandard.net/AboutUs.aspx>

Contact: Wayne Zheng, Sales@ChineseStandard.net

Linkin: <https://www.linkedin.com/in/waynezhengwenrui/>

----- The End -----