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National Food Safety Standard

Food Contact Materials and Products Determination of Migration of Laurolactam

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

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

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Food Contact Materials and Products

Determination of Migration of Laurolactam

1 Scope

This standard specifies the method for the determination of the migration of laurolactam in food contact materials and products.

This standard applies to the determination of the migration of laurolactam in polyamide food contact materials and products.

Method 1 Liquid Chromatography

2 Principle

After the migration test of food contact materials and products, liquid chromatography is used for detection. Among them, aqueous, acidic, ethanol-containing food simulants and chemical alternative solvent 95% (volume fraction) ethanol were directly injected after filtration; oil-containing food simulants were filtered and injected after liquid-liquid extraction and solid-phase extraction; the chemical alternative solvent isooctane was filtered and injected after removing the solvent and re-dissolving in acetonitrile. Retention time was used for qualitative analysis and external standard method for quantitative analysis.

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 Acidic, ethanolic, oily food simulants and chemically substituted solvents: The reagents used shall comply with the provisions of GB5009.156.

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% to 28%.

3.1.5 Anhydrous ethanol (C_2H_6O).

3.2 Reagent preparation

3.2.1 Formic acid-water solution: Measure 5.0 mL formic acid and 95 mL 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: Take 20 mL of acetonitrile and 80 mL of water, pipette 100 μL of formic acid, and mix well.

3.3 Standards

Laurolactam standard ($C_{12}H_{23}NO$, CAS: 947-04-6, also known as lauryl lactam or azacyclotridecan-2-one): purity ≥ 98%, or standard substances certified by the state and awarded with standard substance certificates.

3.4 Preparation of standard solution

3.4.1 Standard stock solution of laurolactam (1000 mg/L): Weigh 100 mg (accurate to 0.1 mg) of laurolactam standard, dissolve it in anhydrous ethanol, make up to 100 mL, and shake well. Transfer the solution to a brown glass container and store it at 4°C away from light. The shelf life is 3 months.

3.4.2 Standard intermediate solution of laurolactam (200 mg/L): Pipette 5.0 mL of standard stock solution of laurolactam into a 25 mL volumetric flask, dilute to the mark with anhydrous ethanol, and shake well. Transfer the solution to a brown glass container and store it at 4°C away from light. The shelf life is 3 months.

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

containing food simulants or 95% (volume fraction) ethanol to prepare standard working solutions with concentrations of 1.0mg/L, 2.0mg/L, 5.0mg/L, 10mg/L and 20mg/L respectively. Prepare immediately before use.

3.4.4 Standard working solution of oil-containing food simulant: Weigh 2g (accurate to 0.01g) of olive oil, add 20μL, 30μL, 50μL of laurolactam standard intermediate solution and 20μL, 40μL of laurolactam standard stock solution respectively, mix well, and prepare standard working solutions with concentrations of 2.0mg/kg, 3.0mg/kg, 5.0mg/kg, 10mg/kg and 20mg/kg respectively. Prepare immediately before use. Before loading on the machine, treat it simultaneously with the soaking solution of oily food simulant according to 5.1.3.

3.4.5 Isooctane standard working solution: Pipette 0.50mL, 1.0mL, 2.5mL, 5.0mL and 10mL of the laurolactam standard intermediate solution into 100mL volumetric flasks respectively and make up to the mark with isooctane. The concentrations of the standard working solutions are 1.0mg/L, 2.0mg/L, 5.0mg/L, 10mg/L and 20mg/L respectively. Prepare immediately before use. Before loading, treat with isooctane soaking solution according to 5.1.4.

4. Instruments and equipment

4.1 High performance liquid chromatograph: equipped with a diode array detector or ultraviolet detector.

4.2 Adjustable pipette: range 10μL～100μL and range 100μL～1000μL.

4.3 Analytical balance: sensitivity 0.0001g (0.1mg) and sensitivity 0.01g.

4.4 Organic microporous filter membrane: 0.45μm.

4.5 Vortex mixer.

4.6 Centrifuge.

4.7 Nitrogen concentrator.

4.8 Solid phase extraction device.

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

5 Analysis steps

5.1 Preparation of test solution

5.1.1 Migration test

Food contact materials and products are subjected to migration tests in accordance with the requirements of GB5009.156 and GB31604.1 to obtain immersion solutions.

5.1.2 Aqueous, acidic, ethanol-containing food simulants and 95% (volume fraction) ethanol test solution

Take 1mL to 2mL of the aqueous, acidic, ethanol-containing food simulants and 95% (volume fraction) ethanol soaking solution obtained from the migration test, filter them through a microporous filter membrane and then prepare them for determination.

5.1.3 Test solution for oily food simulants

Weigh 2 g (accurate to 0.01 g) of the soaking solution of oily food simulant into a 15 mL centrifuge tube, add 4.0 mL of acetonitrile, vortex and extract for 10 minutes, centrifuge at 8500 r/min for 2 minutes, and take the supernatant. The soaking solution was extracted once again, and the two extracts were combined and concentrated to about 0.5 mL under nitrogen blowing at 45°C. After re-dissolving with 4.0 mL of formic acid-water solution, the solution was transferred to a mixed cationic solid phase extraction column activated with 3.0 mL of acetonitrile, pure water, and formic acid-water solution. The mixture was washed with 3.0 mL of formic acid-water solution and acetonitrile in sequence, and then eluted with 3.0 mL of ammonia-acetonitrile solution. The eluate was blown to dryness with nitrogen at 45°C, 1.0 mL of formic acid-acetonitrile-water solution was added, vortexed for 1 minute to re-dissolve, and filtered through a microporous filter membrane before determination.

5.1.4 Isooctane test solution

1.0 mL of the isooctane soaking solution obtained from the migration test was taken out and blown to dryness with nitrogen at 45°C. 1.0 mL of acetonitrile was added to dissolve the residue and filtered through a microporous filter membrane before determination.

5.1.5 Preparation of blank test solution

Food simulants and chemical substitute solvents that are not in contact with food contact materials and products shall be treated according to 5.1.2, 5.1.3 and 5.1.4 to obtain blank test solutions.

5.2 Instrument reference conditions

The instrument reference conditions are as follows:

a) Chromatographic column: C_{18} column, column length 250mm, column inner diameter 4.6mm, particle size 5μm, or 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: 210nm.

5.3 Preparation of standard curve

Determine the standard working solution according to the instrument reference conditions listed in 5.2. A standard curve is drawn with the concentration of laurolactam in the standard working solution as the abscissa and the corresponding peak area as the ordinate. The reference chromatogram of laurolactam standard solution is shown in Figure A.1 in Annex A.

5.4 Determination of test solution

Determine the food simulant test solution and blank test solution according to the instrument reference conditions listed in 5.2, and obtain the peak area of the target

substance by qualitative analysis based on the retention time. Obtain the concentration of laurolactam in the test solution based on the standard curve. Calculate the migration of laurolactam according to Chapter 6. The retention time of the chromatographic peak of the food simulant test solution should deviate from the retention time of the chromatographic peak of the standard working solution within the range of ±2.5%.

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

6. Presentation of analysis results

6.1 Calculation of the migration of laurolactam in food simulants:

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

$$X_1 = (c - c_0) \cdot N \quad \text{..... (1)}$$

Where:

X_1 ——the migration amount of laurolactam in food simulants, in milligrams per liter (mg/L) or milligrams per kilogram (mg/kg);

c——the content of laurolactam in the food simulant test solution, in milligrams per liter (mg/L) or milligrams per kilogram (mg/kg);

c_0 ——the content of laurolactam in the blank test solution, in milligrams per liter (mg/L) or milligrams per kilogram (mg/kg);

N——dilution multiple.

6.2 Calculation of specific migration of laurolactam in food contact materials and products

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

For food contact materials and products other than sealing products such as lids, sealing rings, connectors (hereinafter referred to as sealing products), the specific

migration amount of laurolactam, when expressed in mg/kg, shall be calculated according to formula (2).

Where:

$$X_2 = X_1 \cdot \frac{V_1 \cdot S_0}{S_1 \cdot m_1} \quad \text{.....(2)}$$

X_2 ——Specific migration amount of laurolactam in food contact materials and products, in milligrams per kilogram (mg/kg);

V_1 ——the volume or mass of the sample immersion solution in the migration test, in liters (L) or kilograms (kg);

S_0——the area of food contact materials and products that are in contact with food during actual use, in square decimeters (dm^2);

S_1 ——the area of contact between the sample and the immersion liquid in the migration test, in square decimeters (dm^2);

m_1 ——The mass of solid food or the mass of liquid food that food contact materials and products are in contact with during actual use, in kilograms (kg); the volume of various liquid foods is converted into the corresponding mass based on the density of 1kg/L.

The result must have at least 2 significant digits.

6.2.2 Calculation of specific migration amount of laurolactam in food contact materials and products of sealing products (expressed in mg/kg)

When the intended use is known, for food contact materials and products such as sealing products, the specific migration amount of laurolactam is expressed in mg/kg and is calculated according to formula (3).

Where:

$$X_3 = X_1 \cdot \frac{V_1 \cdot S_0}{S_1 \cdot m_2} \quad \text{.....(3)}$$

X_3 ——Specific migration amount of laurolactam in food contact materials and products, in milligrams per kilogram (mg/kg);

V_1 ——the volume or mass of the sample immersion solution in the migration test, in liters (L) or kilograms (kg);

S_0 ——the area of the sealing product that is in contact with food during actual use, in square decimeters (dm^2);

S_1 ——the area of contact between the sample and the immersion liquid in the migration test, in square decimeters (dm^2);

m_2——The mass of solid food or the volume of liquid food in the actual supporting container of the sealed product, in kilograms (kg); the volume of various liquid foods is converted into the corresponding mass based on the density of 1kg/L.

The result must have at least 2 significant digits.

6.2.3 Calculation of specific migration amount of laurolactam in food contact materials and products of sealing products (expressed in mg/piece)

When the intended use is unknown, for food contact materials and products such as sealing products, the specific migration amount of laurolactam is expressed in mg/piece and is calculated according to formula (4). The migration test method adopted and the contact area between a single sealing product and the food simulant in the migration test must be indicated.

$$X_4 = X_1 \cdot \frac{V_1}{n} \quad \text{.....(4)}$$

Where:

X_4 ——Specific migration amount of laurolactam in food contact materials and products, in milligrams per piece (mg/piece);

V_1 ——the volume or mass of the sample immersion solution in the migration test, in liters (L) or kilograms (kg);

n——The number of sealing products immersed in the migration test, in pieces.

The result must have at least 2 significant digits.

7. Precision

The absolute difference between two independent determination results obtained under repeatability conditions shall not exceed 10% of the arithmetic mean.

8 Others

The detection limit of laurolactam in aqueous, acidic, ethanol-containing food simulants and chemical alternative solvents 95% (volume fraction) ethanol and isooctane is 0.5 mg/L, and the quantitation limit is 1.0 mg/L. The detection limit of laurolactam in oil-containing simulants is 1.0 mg/kg, and the quantitation limit is 2.0 mg/kg.

The detection limit and quantitation limit of specific migration of laurolactam in food contact materials and products are based on the detection limit and quantitation limit of laurolactam in food simulants and converted in accordance with Chapter 6.

Method 2: 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, aqueous, acidic, ethanol-containing food simulants and chemical substitute solvent 95% (volume fraction) ethanol were directly injected after filtration; oil-containing food simulants were filtered and injected after liquid-liquid extraction and solid-phase extraction; the chemical substitute solvent isooctane was filtered and injected after removing the solvent and re-dissolving in acetonitrile. Retention time and relative ion abundance ratio were used for qualitative analysis, and external standard method was used for quantitative analysis.

10 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.

10.1 Reagents

Same as 3.1.

10.2 Solution preparation

Same as 3.2.

10.3 Standards

Same as 3.3.

10.4 Preparation of standard solution

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

10.4.2 Standard intermediate solution of laurolactam (10 mg/L): Pipette 1 mL of the standard stock solution of laurolactam into a 100 mL volumetric flask, dilute to the mark with anhydrous ethanol, and shake well. Transfer the solution to a brown glass container and store it at 4°C away from light. The shelf life is 3 months.

10.4.3 Standard intermediate solution of laurolactam (1.0 mg/L): Pipette 1 mL of the standard stock solution of laurolactam into a 10 mL volumetric flask, dilute to the mark with anhydrous ethanol, and shake well. Transfer the solution to a brown glass container and store it at 4°C away from light. The shelf life is 3 months.

10.4.4 Aqueous, acidic, ethanol-containing food simulants and 95% (volume fraction) ethanol standard working solutions: Accurately pipette 0.30mL, 0.50mL, 1.0mL, 5.0mL and 10mL of the laurolactam standard intermediate solution (1.0mg/L) into a 100mL volumetric flask, dilute to the mark with the corresponding aqueous, acidic, ethanol-containing food simulants or 95% (volume fraction) ethanol to prepare 0.003mg/L, 0.005mg/L, 0.010mg/L, 0.050mg/L and 0.10mg/L standard working solutions. Prepare immediately before use.

10.4.5 Standard working solution of oily food simulant: pipette 12μL, 20μL, 40μL of laurolactam standard intermediate solution (1.0mg/L) and 10μL, 20μL of laurolactam standard intermediate solution (10mg/L) respectively, add to 2g (accurate to 0.01g) of oily food simulant, mix well, and prepare standard working solutions of 0.006mg/kg, 0.010mg/kg, 0.020mg/kg, 0.050mg/kg, and 0.10mg/kg. Prepare immediately before use. Before loading on the machine, treat it simultaneously with the soaking solution of oily food simulant according to 12.1.

10.4.6 Isooctane standard working solution: Pipette 0.30mL, 0.50mL, 1.0mL, 5.0mL and 10mL of laurolactam standard intermediate solution (1.0mg/L) into a 100mL volumetric flask, dilute to the mark with isooctane. The concentrations of the

standard working solutions are 0.003mg/L, 0.005mg/L, 0.010mg/L, 0.050mg/L and 0.10mg/L, respectively. Prepare immediately before use. Before using the machine, treat it simultaneously with isooctane soaking solution according to 12.1.

11 Instruments and equipment

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

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

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

11.4 Organic microporous filter membrane: 0.45μm.

11.5 Vortex mixer.

11.6 Centrifuge.

11.7 Nitrogen Concentrator.

11.8 Solid phase extraction apparatus.

11.9 Mixed cationic solid phase extraction cartridge or equivalent purification cartridge: 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 liquid chromatography reference conditions are as follows:

a) Chromatographic column: C_{18} chromatographic column, column length 100 mm, column inner diameter 2.1 mm, particle size 1.7 μm, or chromatographic 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:

Table 1 Gradient elution conditions

Time/min	A/%	B/%
0.0	60	40
0.5	60	40
0.6	50	50
4.0	50	50
4.1	2	98
5.2	2	98
5.3	60	40
7.0	60	40

12.2.2 Mass spectrometry conditions

The mass spectrometry reference conditions are as follows:

a) Ionization mode: electrospray ionization positive mode (ESI^+);

b) Mass spectrometry scanning mode: multiple reaction monitoring (MRM);

c) Spray voltage: 5500V;

d) Atomization temperature: 500°C;

e) The mass spectrometry conditions of laurolactam refer to Table 2.

Table 2 Reference conditions for laurolactam mass spectrometry

Compound	Precursor ion (m/z)	Product ion (m/z)	Fragmentation	Collision energy/eV

			voltage/V	
laurolactam	198.2	83.1 [a]	80	28
	198.2	97.0	80	28
Note: Instrument parameters may vary for different mass spectrometers, and mass spectrometer parameters should be optimized before determination.				
[a] is the quantitative ion.				

12.3 Qualitative determination

Determine the food simulant test solution and standard working solution under the instrument reference conditions listed in 12.2. The following conditions must be met to determine the presence of the corresponding target substance in the test solution:

a) The difference between the retention time of the chromatographic peak of the target compound in the test solution and the retention time of the chromatographic peak of the target compound in the standard working solution is within ±2.5%;

b) The signals of the two selected ion pairs appear;

c) The deviation between the relative abundance of the qualitative ion pair and the relative abundance of the standard solution with equivalent concentration shall not exceed the provisions in Table 3.

Table 3 Maximum allowable deviation of relative ion abundance in qualitative determination

Relative ion abundance/%	>50	20～50	10～20	10
Allowable relative error/%	± 20	± 25	± 30	± 50

12.4 Preparation of standard curve

Determine the standard working solution using the instrument reference conditions listed in 12.2. A standard curve is drawn with the concentration of laurolactam in the standard working solution as the abscissa and the corresponding peak area as the ordinate. The reference chromatogram of the multiple reaction monitoring (MRM) of laurolactam standard solution is shown in Figure A.2 in Annex A.

12.5 Quantitative determination

Determine the food simulant test solution and blank test solution according to the instrument reference conditions listed in 12.2 to obtain the peak area of the target substance and obtain the concentration of laurolactam in the test solution based on the standard curve. Calculate the migration of laurolactam according to Chapter 13.

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

13 Presentation of analysis results

Same as Chapter 6.

14 Precision

The absolute difference between two independent determination results obtained under repeatability conditions shall not exceed 10% of the arithmetic mean.

15 Others

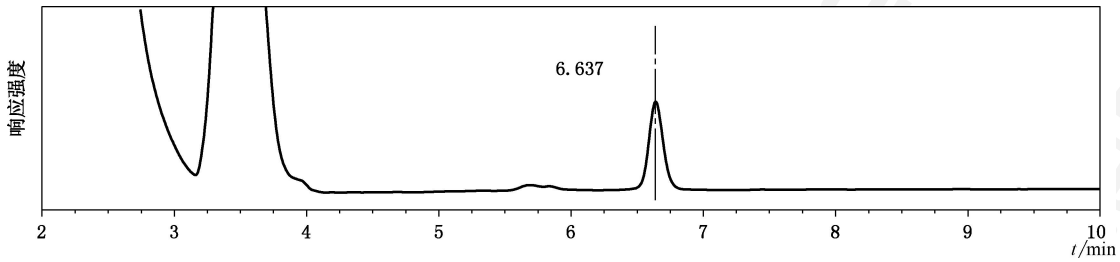
The detection limit of laurolactam in aqueous, acidic, ethanol-containing food simulants and 95% (volume fraction) ethanol and isooctane food simulants is 0.001 mg/L, and the quantitation limit is 0.003 mg/L. The detection limit of laurolactam in oil -containing food simulants is 0.002 mg/kg, and the quantitation limit is 0.006 mg/kg.

The detection limit and quantitation limit of specific migration of laurolactam in food contact materials and products are based on the detection limit and quantitation limit of laurolactam in food simulants and converted in accordance with Chapter 13 .

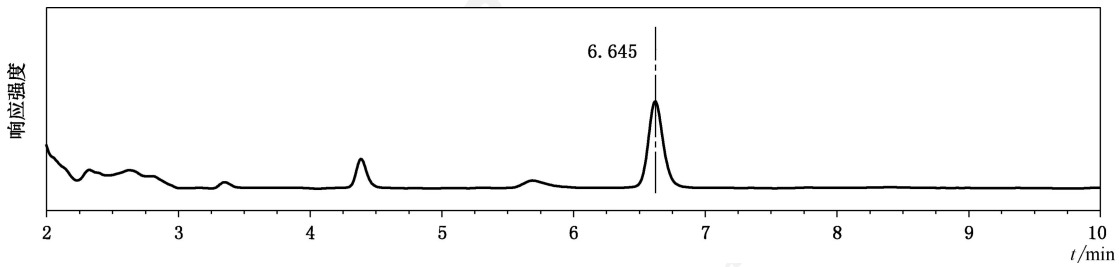
Annex A

Chromatogram of laurolactam standard solution

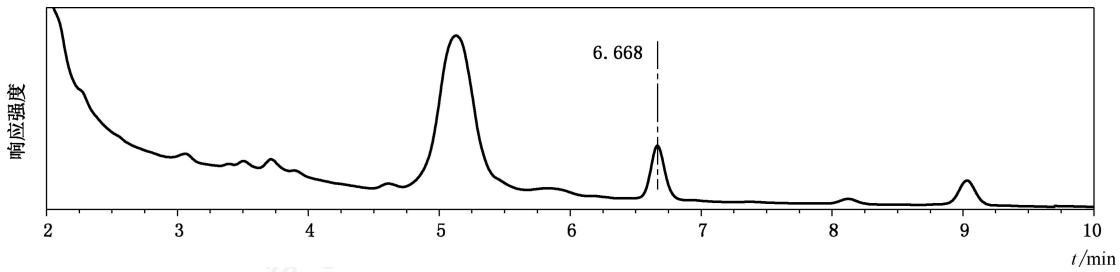
The liquid chromatogram of laurolactam standard solution in different food simulants is shown in Figure A.1, and the liquid chromatography-tandem mass spectrometry MRM diagram is shown in Figure A.2 .



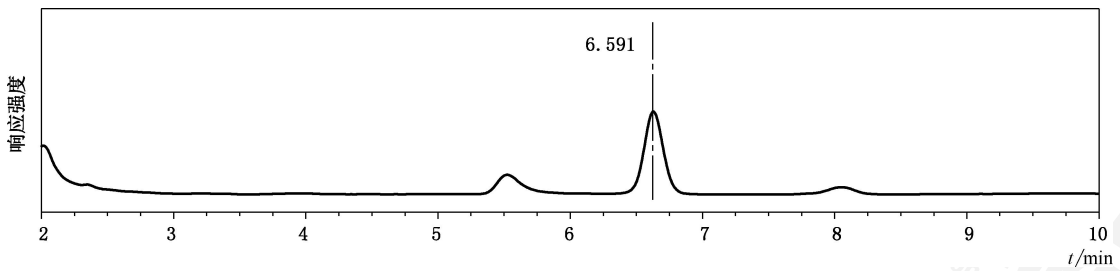
a) 4% acetic acid



b) 50% ethanol



c) Olive oil



d) Isooctane

Figure A.1 Liquid chromatograms of laurolactam standard solutions (5 mg /L or 5 mg / kg) in different food simulants and chemical alternative solvents

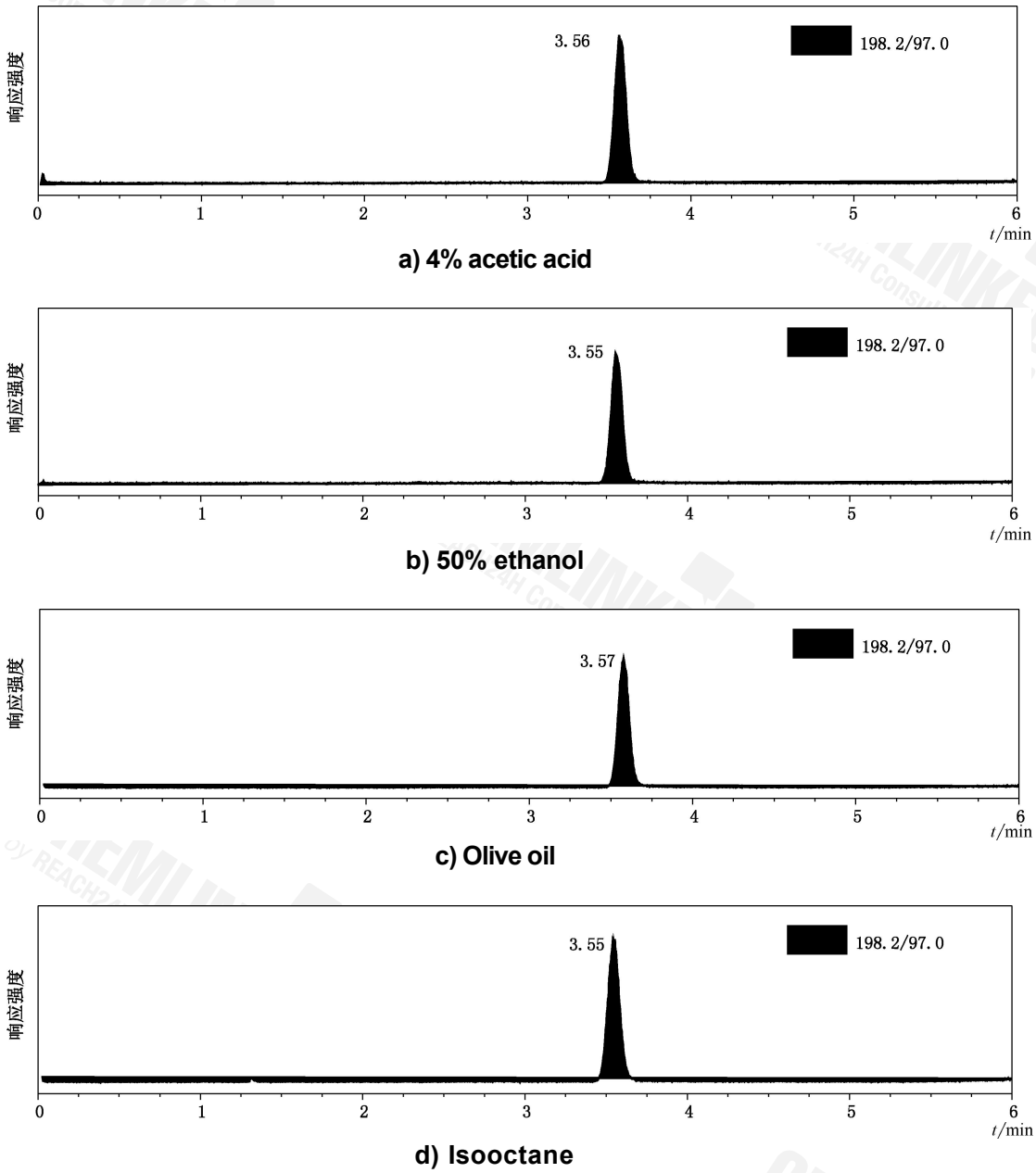


Figure A.2 MRM diagram of laurolactam standard solution (0.05 mg /L or 0.05 mg / kg) in different food simulants and chemical alternative solvents

中华人民共和国国家标准

GB 31604.56—2023

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

2023-09-06 发布　　2024-03-06 实施

中华人民共和国国家卫生健康委员会
国家市场监督管理总局　发布

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

1 范围

本标准规定了食品接触材料及制品中月桂内酰胺迁移量的测定方法。

本标准适用于聚酰胺类食品接触材料及制品中月桂内酰胺迁移量的测定。

第一法 液相色谱法

2 原理

食品接触材料及制品进行迁移试验后，采用液相色谱检测。其中水性、酸性、含乙醇食品模拟物及化学替代溶剂95%(体积分数)乙醇经过滤后直接进样；含油脂食品模拟物经液液萃取和固相萃取后过滤进样；化学替代溶剂异辛烷通过去除溶剂和乙腈复溶后过滤进样。保留时间定性，外标法定量。

3 试剂和材料

除非另有说明，本方法所用试剂均为分析纯，水为GB/T 6682规定的一级水。

3.1 试剂

3.1.1 酸性、含乙醇、含油脂食品模拟物和化学替代溶剂：所用试剂按照GB 5009.156的规定。

3.1.2 乙腈(C_2H_3N)：色谱纯。

3.1.3 甲酸(CH_2O_2)：色谱纯。

3.1.4 氨水：氨的质量分数为25%～28%。

3.1.5 无水乙醇(C_2H_6O)。

3.2 试剂配制

3.2.1 甲酸-水溶液：量取5.0 mL甲酸和95 mL水，混匀。

3.2.2 氨水-乙腈溶液：量取2.0 mL氨水、8.0 mL水和90 mL乙腈，混匀。

3.2.3 甲酸-乙腈-水溶液：量取20 mL乙腈和80 mL水，吸取100 μL甲酸，混匀。

3.3 标准品

月桂内酰胺标准品($C_{12}H_{23}NO$，CAS：947-04-6，又称十二内酰胺或氮杂环十三烷-2-酮)：纯度≥98%，或经国家认证并授予标准物质证书的标准物质。

3.4 标准溶液配制

3.4.1 月桂内酰胺标准储备溶液(1 000 mg/L)：称取100 mg(精确至0.1 mg)月桂内酰胺标准品，用

无水乙醇溶解后定容至 100 mL，摇匀。将溶液转移至棕色玻璃容器中，于 4 ℃避光保存，有效期3 个月。

3.4.2 月桂内酰胺标准中间溶液(200 mg/L)：移取 5.0 mL 月桂内酰胺标准储备溶液至 25 mL 容量瓶中，用无水乙醇定容至刻度，摇匀。将溶液转移至棕色玻璃容器中，于 4 ℃避光保存，有效期 3 个月。

3.4.3 水性、酸性、含乙醇食品模拟物或 95%(体积分数)乙醇标准工作溶液：分别移取月桂内酰胺标准中间溶液 0.50 mL、1.0 mL、2.5 mL、5.0 mL、10 mL 至 100 mL 容量瓶中，用相应水性、酸性、含乙醇食品模拟物或 95%(体积分数)乙醇定容至刻度，配制浓度分别为 1.0 mg/L、2.0 mg/L、5.0 mg/L、10 mg/L和 20 mg/L 的标准工作溶液。临用现配。

3.4.4 含油脂食品模拟物标准工作溶液：称取 2 g(精确至 0.01 g)橄榄油，分别加入 20 μL、30 μL、50 μL月桂内酰胺标准中间溶液和 20 μL、40 μL 月桂内酰胺标准储备溶液，混匀，配制成浓度分别为 2.0 mg/kg、3.0 mg/kg、5.0 mg/kg、10 mg/kg 和 20 mg/kg 的标准工作溶液。临用现配。上机前按照 5.1.3 与含油脂食品模拟物浸泡液同步处理。

3.4.5 异辛烷标准工作溶液：分别移取月桂内酰胺标准中间溶液 0.50 mL、1.0 mL、2.5 mL、5.0 mL、10 mL至 100 mL 容量瓶中，用异辛烷定容至刻度，标准工作溶液的浓度分别为 1.0 mg/L、2.0 mg/L、5.0 mg/L、10 mg/L 和 20 mg/L。临用现配。上机前按照 5.1.4 与异辛烷浸泡液同步处理。

4 仪器和设备

4.1 高效液相色谱仪：配有二极管阵列检测器或紫外检测器。

4.2 可调移液器：量程 10 μL～100 μL 和量程 100 μL～1 000 μL。

4.3 分析天平：感量 0.000 1 g(0.1 mg)和感量 0.01 g。

4.4 有机微孔滤膜：0.45 μm。

4.5 涡旋混合器。

4.6 离心机。

4.7 氮气浓缩装置。

4.8 固相萃取装置。

4.9 混合型阳离子固相萃取小柱或等效净化柱：150 mg/6 mL。

5 分析步骤

5.1 试液的制备

5.1.1 迁移试验

食品接触材料及制品按照 GB 5009.156 和 GB 31604.1 的要求开展迁移试验，得到浸泡液。

5.1.2 水性、酸性、含乙醇食品模拟物和 95%(体积分数)乙醇试液

移取 1 mL～2 mL 迁移试验得到的水性、酸性、含乙醇食品模拟物和 95%(体积分数)乙醇浸泡液，通过微孔滤膜过滤后供测定。

5.1.3 含油脂食品模拟物试液

称取 2 g(精确到 0.01 g)含油脂食品模拟物浸泡液于 15 mL 离心管中，加入 4.0 mL 乙腈，涡旋振荡提取 10 min，以 8 500 r/min 离心 2 min，取上清液。重复提取浸泡液一次，合并两次提取液，在 45 ℃下氮吹浓缩至约 0.5 mL。用 4.0 mL 甲酸-水溶液复溶后，转移至用 3.0 mL 乙腈、纯水、甲酸-水溶液活化

的混合型阳离子固相萃取小柱。用 3.0 mL 甲酸-水溶液、乙腈依次淋洗，用 3.0 mL 氨水-乙腈溶液洗脱。洗脱液在 45 ℃下氮吹至干，加入 1.0 mL 甲酸-乙腈-水溶液，涡旋 1 min 复溶，微孔滤膜过滤后供测定。

5.1.4 异辛烷试液

移取 1.0 mL 迁移试验得到的异辛烷浸泡液，45 ℃下用氮气吹至干，加入 1.0 mL 乙腈溶解残渣，微孔滤膜过滤后供测定。

5.1.5 空白试液的制备

未与食品接触材料及制品接触的食品模拟物和化学替代溶剂按 5.1.2、5.1.3 和 5.1.4 处理，得到空白试液。

5.2 仪器参考条件

仪器参考条件如下：

a) 色谱柱：C_{18}柱，柱长 250 mm，柱内径 4.6 mm，粒径 5 μm，或者同等性能的色谱柱；
b) 流动相：乙腈和水(50+50，体积比)；
c) 流速：1.0 mL/min；
d) 进样量：20 μL；
e) 柱温：25 ℃；
f) 检测波长：210 nm。

5.3 标准曲线的制作

按照 5.2 所列的仪器参考条件测定标准工作溶液。以标准工作溶液中月桂内酰胺的浓度为横坐标，以对应的峰面积为纵坐标，绘制标准曲线。月桂内酰胺标准溶液的参考色谱图见附录 A 中图 A.1。

5.4 试液的测定

按照 5.2 所列的仪器参考条件测定食品模拟物试液和空白试液，以保留时间定性，得到目标物峰面积，根据标准曲线得到试液中月桂内酰胺的浓度。按第 6 章计算月桂内酰胺迁移量。食品模拟物试液色谱峰的保留时间与标准工作溶液色谱峰的保留时间偏差应在±2.5%范围内。

试液可根据具体情况稀释，使其测定值在标准曲线的线性范围内。

6 分析结果的表述

6.1 食品模拟物中月桂内酰胺迁移量的计算：

食品模拟物中月桂内酰胺的迁移量按式(1)计算。

$$X_1 = (c - c_0) \cdot N \qquad \cdots\cdots(1)$$

式中：

X_1——食品模拟物中月桂内酰胺的迁移量，单位为毫克每升(mg/L)或毫克每千克(mg/kg)；

c ——食品模拟物试液中月桂内酰胺的含量，单位为毫克每升(mg/L)或毫克每千克(mg/kg)；

c_0 ——空白试液中月桂内酰胺的含量，单位为毫克每升(mg/L)或毫克每千克(mg/kg)；

N ——稀释倍数。

6.2 食品接触材料及制品中月桂内酰胺特定迁移量的计算

6.2.1 非密封制品类食品接触材料及制品月桂内酰胺特定迁移量的计算(以 mg/kg 表示)

除了盖子、密封圈、连接件等密封制品(以下简称密封制品)以外的食品接触材料及制品,月桂内酰胺特定迁移量以 mg/kg 表示时,按式(2)计算。

$$X_2 = X_1 \cdot \frac{V_1 \cdot S_0}{S_1 \cdot m_1} \quad \cdots\cdots(2)$$

式中:

X_2——食品接触材料及制品中月桂内酰胺的特定迁移量,单位为毫克每千克(mg/kg);

V_1——迁移试验中试样浸泡液的体积或质量,单位为升(L)或千克(kg);

S_0——食品接触材料及制品实际使用时接触食品的面积,单位为平方分米(dm^2);

S_1——迁移试验中试样与浸泡液接触的面积,单位为平方分米(dm^2);

m_1——食品接触材料及制品实际使用时接触固态食品质量或液体食品体积对应的质量,单位为千克(kg);各种液态食品按密度为 1 kg/L 将体积换算为对应的质量。

结果至少保留 2 位有效数字。

6.2.2 密封制品类食品接触材料及制品月桂内酰胺特定迁移量的计算(以 mg/kg 表示)

当预期用途已知时,密封制品类食品接触材料及制品,月桂内酰胺特定迁移量以 mg/kg 表示时,按式(3)计算。

$$X_3 = X_1 \cdot \frac{V_1 \cdot S_0}{S_1 \cdot m_2} \quad \cdots\cdots(3)$$

式中:

X_3——食品接触材料及制品中月桂内酰胺的特定迁移量,单位为毫克每千克(mg/kg);

V_1——迁移试验中试样浸泡液的体积或质量,单位为升(L)或千克(kg);

S_0——密封制品实际使用时接触食品的面积,单位为平方分米(dm^2);

S_1——迁移试验中试样与浸泡液接触的面积,单位为平方分米(dm^2);

m_2——密封制品实际配套使用容器盛装固态食品质量或液体食品体积对应的质量,单位为千克(kg);各种液态食品按密度为 1 kg/L 将体积换算为对应的质量。

结果至少保留 2 位有效数字。

6.2.3 密封制品类食品接触材料及制品月桂内酰胺特定迁移量的计算(以 mg/件表示)

当预期用途未知时,密封制品类食品接触材料及制品,月桂内酰胺特定迁移量以 mg/件表示时,按式(4)计算,需注明采用的迁移试验方法、迁移试验中单个密封制品与食品模拟物接触的面积。

$$X_4 = X_1 \cdot \frac{V_1}{n} \quad \cdots\cdots(4)$$

式中:

X_4——食品接触材料及制品中月桂内酰胺的特定迁移量,单位为毫克每件(mg/件);

V_1——迁移试验中试样浸泡液的体积或质量,单位为升(L)或千克(kg);

n——迁移试验中浸泡用密封制品的数量,单位为件。

结果至少保留 2 位有效数字。

7 精密度

在重复性条件下获得的两次独立测定结果的绝对差值不得超过算术平均值的10%。

8 其他

水性、酸性、含乙醇食品模拟物及化学替代溶剂95%(体积分数)乙醇与异辛烷中月桂内酰胺方法检出限为0.5 mg/L,定量限为1.0 mg/L。含油脂模拟物中月桂内酰胺方法检出限为1.0 mg/kg,定量限为2.0 mg/kg。

食品接触材料及制品中月桂内酰胺特定迁移量的检出限与定量限,以食品模拟物中月桂内酰胺方法检出限与定量限为基础,按第6章换算。

第二法 液相色谱-串联质谱法

9 原理

食品接触材料及制品进行迁移试验后,采用液相色谱-串联质谱检测。其中水性、酸性、含乙醇食品模拟物及化学替代溶剂95%(体积分数)乙醇经过滤后直接进样;含油脂食品模拟物经液液萃取和固相萃取后过滤进样;化学替代溶剂异辛烷通过去除溶剂和乙腈复溶后过滤进样。保留时间和相对离子丰度比定性,外标法定量。

10 试剂和材料

除非另有说明,本方法所用试剂均为分析纯,水为GB/T 6682规定的一级水。

10.1 试剂

同3.1。

10.2 溶液配制

同3.2。

10.3 标准品

同3.3。

10.4 标准溶液配制

10.4.1 月桂内酰胺标准储备溶液(1 000 mg/L):同3.4.1。

10.4.2 月桂内酰胺标准中间溶液(10 mg/L):移取月桂内酰胺标准储备溶液1 mL于100 mL容量瓶中,用无水乙醇定容至刻度,摇匀。将溶液转移至棕色玻璃容器中,于4 ℃避光保存,有效期3个月。

10.4.3 月桂内酰胺标准中间溶液(1.0 mg/L):移取月桂内酰胺标准储备溶液1 mL于10 mL容量瓶中,用无水乙醇定容至刻度,摇匀。将溶液转移至棕色玻璃容器中,于4 ℃避光保存,有效期3个月。

10.4.4 水性、酸性、含乙醇食品模拟物及95%(体积分数)乙醇标准工作溶液:分别准确移取0.30 mL、

0.50 mL、1.0 mL、5.0 mL 和 10 mL 月桂内酰胺标准中间溶液(1.0 mg/L)至 100 mL 容量瓶中,用相应水性、酸性、含乙醇食品模拟物或 95%(体积分数)乙醇定容至刻度,配制成 0.003 mg/L、0.005 mg/L、0.010 mg/L、0.050 mg/L、0.10 mg/L 的标准工作溶液。临用现配。

10.4.5 含油脂食品模拟物标准工作溶液:分别移取 12 μL、20 μL、40 μL 月桂内酰胺标准中间溶液(1.0 mg/L)和 10 μL、20 μL 月桂内酰胺标准中间溶液(10 mg/L),加入到 2 g(精确到 0.01 g)含油脂食品模拟物中,混匀,配制成 0.006 mg/kg、0.010 mg/kg、0.020 mg/kg、0.050 mg/kg、0.10 mg/kg 的标准工作溶液。临用现配。上机前按照 12.1 与含油脂食品模拟物浸泡液同步处理。

10.4.6 异辛烷标准工作溶液:分别移取 0.30 mL、0.50 mL、1.0 mL、5.0 mL 和 10 mL 月桂内酰胺标准中间溶液(1.0 mg/L)至 100 mL 容量瓶中,用异辛烷定容至刻度,标准工作溶液的浓度分别为 0.003 mg/L、0.005 mg/L、0.010 mg/L、0.050 mg/L、0.10 mg/L。临用现配。上机前按照 12.1 与异辛烷浸泡液同步处理。

11 仪器和设备

11.1 液相色谱-串联质谱仪:配备电喷雾离子源(ESI)。

11.2 可调移液器:量程为 10 μL～100 μL 和量程为 100 μL～1 000 μL。

11.3 分析天平:感量 0.000 1 g(0.1 mg)和感量 0.01 g。

11.4 有机微孔滤膜:0.45 μm。

11.5 涡旋混合器。

11.6 离心机。

11.7 氮气浓缩装置。

11.8 固相萃取装置。

11.9 混合型阳离子固相萃取小柱或等效净化柱:150 mg/6 mL。

12 分析步骤

12.1 样品前处理

同 5.1。

12.2 仪器参考条件

12.2.1 液相色谱条件

液相色谱参考条件如下:

a) 色谱柱:C_{18}色谱柱,柱长 100 mm,柱内径 2.1 mm,粒径 1.7 μm,或者同等性能的色谱柱;

b) 流动相:A 为含 0.1%(体积分数)甲酸的水溶液,B 为含 0.1%(体积分数)甲酸的乙腈溶液,洗脱梯度见表 1;

c) 流速:0.3 mL/min;

d) 柱温:40 ℃;

e) 进样量:2 μL。

表 1　梯度淋洗条件

时间/min	A/%	B/%
0.0	60	40
0.5	60	40
0.6	50	50
4.0	50	50
4.1	2	98
5.2	2	98
5.3	60	40
7.0	60	40

12.2.2　质谱条件

质谱参考条件如下：

a)　离子化模式：电喷雾电离正离子模式(ESI^+)；

b)　质谱扫描方式：多反应监测(MRM)；

c)　喷雾电压：5 500 V；

d)　雾化温度：500 ℃；

e)　月桂内酰胺质谱条件参考表 2。

表 2　月桂内酰胺质谱参考条件

化合物	母离子(m/z)	子离子(m/z)	碎裂电压/V	碰撞能量/eV
月桂内酰胺	198.2	83.1[a]	80	28
	198.2	97.0	80	28
注：对于不同质谱仪器，仪器参数可能存在差异，测定前应优化质谱参数。				
[a] 为定量离子。				

12.3　定性测定

按照 12.2 所列仪器参考条件测定食品模拟物试液和标准工作溶液，判定试液中存在相应目标物应满足以下条件：

a)　试液中目标化合物色谱峰保留时间与标准工作溶液中目标化合物色谱峰保留时间差异在±2.5%范围内；

b)　选择的两组离子对的信号均出现；

c)　定性离子对的相对丰度与浓度相当的标准溶液的相对丰度偏差不超过表 3 的规定。

表 3　定性测定时相对离子丰度的最大允许偏差

相对离子丰度/%	>50	20～50	10～20	≤10
允许相对误差/%	±20	±25	±30	±50

12.4 标准曲线的制作

按照 12.2 所列的仪器参考条件测定标准工作溶液。以标准工作溶液中月桂内酰胺的浓度为横坐标，以对应峰面积为纵坐标，绘制标准曲线。月桂内酰胺标准溶液多反应监测(MRM)参考色谱图见附录 A 中图 A.2。

12.5 定量测定

按照 12.2 所列的仪器参考条件测定食品模拟物试液和空白试液，得到目标物峰面积，根据标准曲线得到试液中月桂内酰胺的浓度。按第 13 章计算月桂内酰胺迁移量。

试液可根据具体情况稀释，使其测定值在标准曲线的线性范围内。

13 分析结果的表述

同第 6 章。

14 精密度

在重复性条件下获得的两次独立测定结果的绝对差值不得超过算术平均值的 10%。

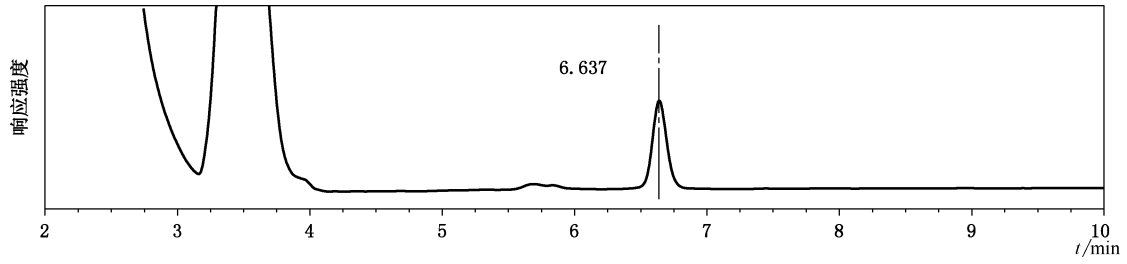
15 其他

水性、酸性、含乙醇食品模拟物及 95%(体积分数)乙醇和异辛烷食品模拟物中月桂内酰胺的检出限为 0.001 mg/L，定量限为 0.003 mg/L。含油脂食品模拟物中月桂内酰胺的检出限为 0.002 mg/kg，定量限为 0.006 mg/kg。

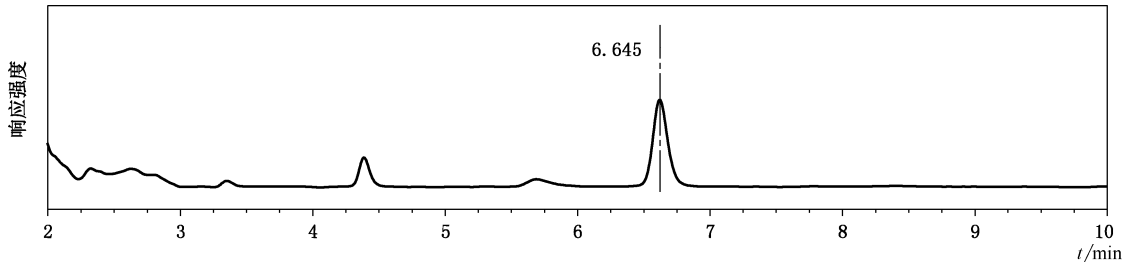
食品接触材料及制品中月桂内酰胺特定迁移量的检出限与定量限，以食品模拟物中月桂内酰胺方法检出限与定量限为基础，按第 13 章换算。

附 录 A
月桂内酰胺标准溶液的色谱图

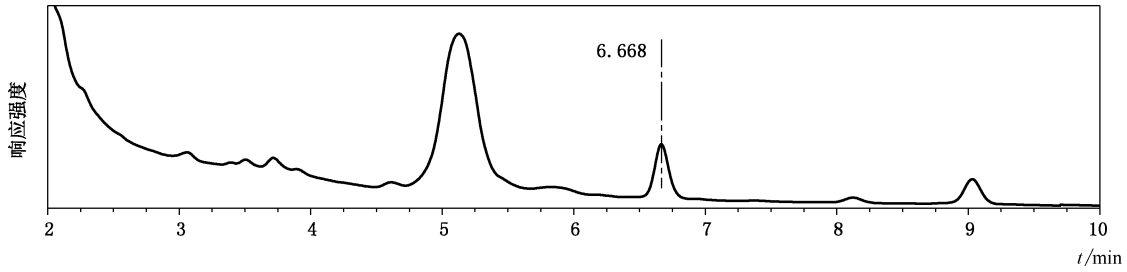
不同食品模拟物中月桂内酰胺标准溶液的液相色谱图见图 A.1，液相色谱-串联质谱法 MRM 图见图 A.2。



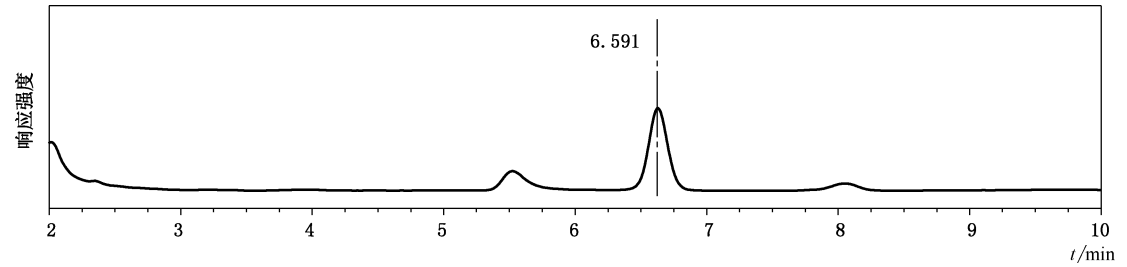
a） 4%乙酸



b） 50%乙醇

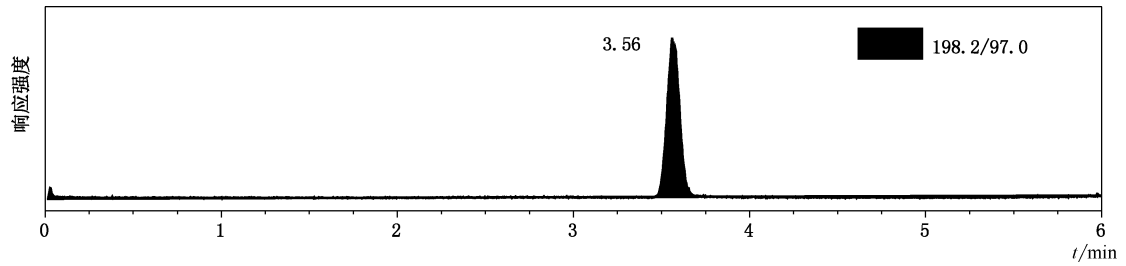


c） 橄榄油

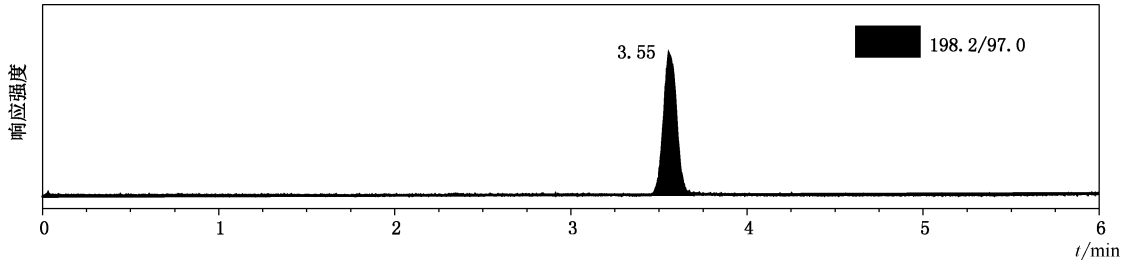


d） 异辛烷

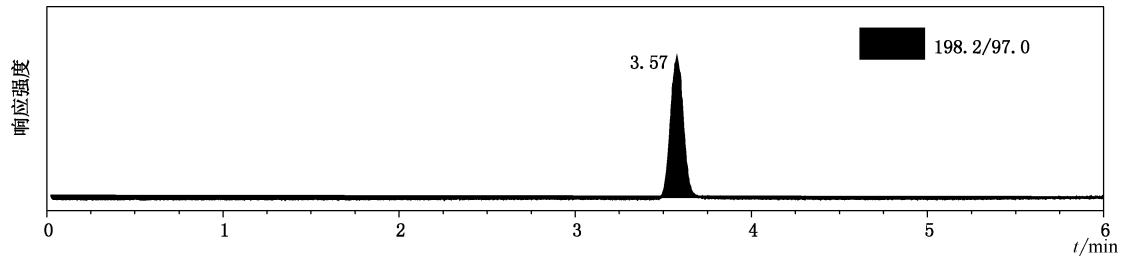
图 A.1 不同食品模拟物和化学替代溶剂中月桂内酰胺标准溶液(5 mg/L 或 5 mg/kg)的液相色谱图



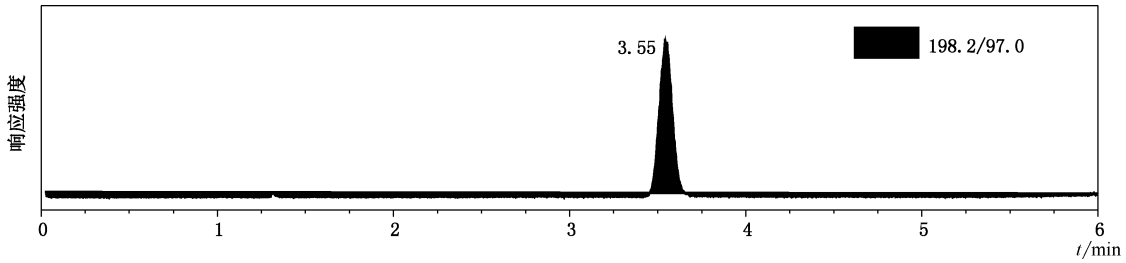
a） 4%乙酸



b） 50%乙醇



c） 橄榄油



d） 异辛烷

图 A.2 不同食品模拟物和化学替代溶剂中月桂内酰胺标准溶液(0.05 mg/L 或 0.05 mg/kg)的 MRM 图

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