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

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1 Scope

This standard specifies determination method of caprolactam and migration quantity in food contact materials and products.

This standard applies to determination of caprolactam and migration quantity in food contact materials and products.

Determination of caprolactam

2 Principles

After the sample is extracted by water, the caprolactam is dissolved in extracting solution; after filtered through filter membrane, it is detected by a high-performance liquid chromatograph (HPLC) that is equipped with an ultraviolet detector; use external standard method to quantify.

3 Reagents and materials

Unless otherwise indicated, the reagents used in this method are analytical grade, the water is the grade-1 water specified in GB/T 6682.

3.1 Reagents

Acetonitrile (C_2H_3N, CAS No: 75-05-8): Chromatographically pure.

3.2 Standards

3.2.1 Caprolactam ($C_6H_{11}NO$, CAS number: 105-60-2): Purity ≥ 99%, or standard substance which is identified by national authentication and awarded a reference material certificate.

3.3 Standard solution preparation

5.2 Preparation of sample solution

WEIGH 1.0 g (accurate to 0.0001g) of crushed samples; PLACE it into a stoppered glass tube; after adding 10 mL of water, EXTRACT for 40 min in a boiling water bath; when cooling to room temperature, TAKE the supernatant into 25 mL volumetric flask. Then USE 10 mL of water to extract once again as above; COMBINE the twice-supernatant; USE water to dilute it to the mark; TAKE 1 mL of extracting solution through 0.45 μm filter membrane; after filtering, it is for sample injection of high performance liquid chromatography. If the concentration of the sample solution is beyond linear range, use water to dilute it so that the concentration is within linear range.

5.3 Preparation of blank solution

Except for not adding sample, use the same analytical procedures, reagents and dosage as 5.2.

5.4 Reference conditions of chromatography

5.4.1 Chromatographic column: C18-column, column-length 250 mm, inner-diameter 4.6 mm, particle-size 5 μm.

5.4.2 Detector: UV detection wavelength 210 nm.

5.4.3 Mobile phase: acetonitrile-water (20+80).

5.4.4 Flow rate: 1.0 mL/min.

5.4.5 Injection volume: 10 μL.

5.5 Drawing of standard curve

According to the measurement conditions listed in 5.4, respectively PLACE caprolactam standard working solution into liquid chromatograph to determine. With the caprolactam concentration of standard working solution as abscissa, in milligrams per liter (mg/L), AND the corresponding peak area of caprolactam as vertical axis, DRAW a standard working curve. See Figure A.1 for the standard chromatogram.

5.6 Determination of sample solution

According to the conditions listed in 5.4, PLACE the sample solution (5.2) and the blank solution (5.3) into liquid chromatograph to determine; OBTAIN the peak area of caprolactam; DEDUCT blank value.

simulant test solution can not be immediately carried out the next test, the food simulant test solution shall be stored in refrigerator at 4 °C.

The resulting food simulant test solution shall be cooled or returned to room temperature before proceeding to the next test.

12.2 Preparation of sample solution

12.2.1 Processing of food simulants that are water-based, acidic and alcoholic

USE graduated pipet to pipette 1 mL of food simulants that are water-based, acidic and alcoholic obtained from the migration test; FILTER through 0.45 μm microporous membrane to wait for determining.

12.2.2 Processing of oil-based food simulants

WEIGH 8.0 g (accurate to 0.01g) of olive oil obtained from the migration test; PLACE it into separatory funnel; ADD 1.6 mL of water; MIX it uniformly; ADD 15 mL of n-hexane; MIX it uniformly; ADD 8 mL of ethanol-water solution (1+2); VIBRATE it for 10 min; LET it stand for 30 min to separate the two-phase; MOVE 5 mL of lower-layer water solution; FILTER through cotton wool and 0.45 μm microporous membrane to wait for determining.

12.3 Preparation of blank solution

Except for not adding sample, use the same analytical procedures, reagents and dosage as 12.2.

12.4 Reference conditions of chromatography

12.4.1 Chromatographic column: C18-column, column-length 250 mm, inner-diameter 4.6 mm, particle-size 5 μm.

12.4.2 Detector: UV detection wavelength 210 nm.

12.4.3 Mobile phase: acetonitrile-water (20+80).

12.4.4 Flow rate: 1.0 mL/min.

12.4.5 Injection volume: 10 μL.

12.5 Drawing of standard curve

According to the conditions listed in 12.4, respectively PLACE standard working solution of food simulants that are water-based, acidic and alcoholic, and standard working solution of oil-based food simulants into liquid chromatograph to determine. With the caprolactam concentration of food simulants as abscissa,

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