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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 31604.13-2016

**National food safety standard -
Food contact materials and products -
Determination of 11-amino undecanoic acid
migration quantity**

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Foreword

This standard replaces GB/T 23296.12-2009 "Food contact materials – Polymer - Determination of 11-aminoundecanoic acid in food simulants - High performance liquid chromatography"

Compared with GB/T 23296.12-2009, the main changes of this standard are as follows:

- The standard name is changed to "National food safety standard - Food contact materials and products - Determination of 11-amino undecanoic acid migration quantity";
- Modify the preparation of food simulant test solution;
- Modify the expression of analysis results.

National food safety standard -

Food contact materials and products - Determination of 11-amino undecanoic acid migration quantity

1 Scope

This standard specifies the test method of high performance liquid chromatography of 11-amino undecanoic acid migration quantity in food contact materials and products.

This standard applies to the determination of 11-amino undecanoic acid migration quantity in food contact materials and products.

2 Principles

11-amino undecanoic acid in food simulants reacts with fluorescamine to form derivative, which is separated by high performance liquid chromatography; use fluorescence detector to test the derivative content; obtain the content of 11-amino undecanoic acid by conversion; 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. The containers and transfer apparatuses in the test shall avoid using plastic materials.

3.1 Reagents

3.1.1 Disodium tetraborate decahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$).

3.1.2 Phosphoric acid (H_3PO_4).

3.1.3 Boric acid (H_3BO_3).

3.1.4 Fluorescent amine ($\text{C}_{17}\text{H}_{10}\text{O}_4$).

3.1.5 Sodium hydroxide (NaOH).

11-amino undecanoic acid ($C_{11}H_{23}O_2N$, CAS No.: 2432-99-7), purity $\geq 99\%$, or standard substance which is identified by national authentication and awarded a reference material certificate.

3.4 Standard solution preparation

3.4.1 11-amino undecanoic acid aqueous-solution stock solution (1.25mg/mL): Accurately WEIGH 125mg (accurate to 0.1mg) of 11-amino undecanoic acid standards; USE 2mL of 96% acetic acid to dissolve it; TRANSFER it into 100mL volumetric flask; USE water to dilute it to the mark; STORE it in a refrigerator at 4 °C.

3.4.2 11-amino undecanoic acid isooctane stock solution (0.50mg/mL): Accurately WEIGH 25mg of 11-amino undecanoic acid standards (accurate to 0.1mg); USE 5mL of glacial acetic acid to dissolve it; TRANSFER it into 50mL volumetric flask; USE isooctane to dilute it to the mark; STORE it in a refrigerator at 4 °C.

3.4.3 11-amino undecanoic acid aqueous-solution standard intermediate solution (25 μ g/mL): Respectively MOVE 2mL of aqueous solution stock solution into 3 different 100mL volumetric flasks; USE water-based, acidic, alcoholic food simulants to dilute it, respectively.

3.4.4 Water-based, acidic food, alcoholic food simulants standard working solution: Respectively MOVE 0mL, 1.0mL, 2.0mL, 4.0mL, 10.0mL, 20.0mL of 11-amino undecanoic acid aqueous solution standard intermediate solution into 50mL volumetric flask; USE water to dilute it. The concentration of the standard working solution is 0 μ g/mL, 0.5 μ g/mL, 1.0 μ g/mL, 2.0 μ g/mL, 5.0 μ g/mL and 10.0 μ g/mL, respectively. In the same way, USE the corresponding acidic and alcoholic food simulants to prepare 11-amino undecanoic acid standard working solution with the same concentration series. PREPARE it when in use.

3.4.5 Oil-based food simulant standard working solution: WEIGH 5g (accurate to 0.01g) of olive oil respectively; PLACE it into 6 different 25mL conical flasks. Respectively ADD 0 μ L, 5 μ L, 10 μ L, 20 μ L, 50 μ L and 100 μ L of 11-amino undecanoic acid isooctane stock solution; MIX it uniformly. The concentration of standard working solution is 0 μ g/g, 0.5 μ g/g, 1.0 μ g/g, 2.0 μ g/g, 5.0 μ g/g and 10.0 μ g/g, respectively. Then operate according to 5.2.2.

4 Instrument and equipment

4.1 High performance liquid chromatography: Equipped with fluorescence detector.

4.2 Oscillator: Reciprocating-type.

Accurately MOVE 1.0mL of boric acid buffer solution into the prepared water-based, acidic, alcoholic, oil-based food simulant test solution and the prepared blank solution; PLACE it on vortex oscillator for 10s; MIX it uniformly. During the mixing process, ADD 1.0mL of fluorescamine solution. The resulting derivative solution is passed through microfiltration membrane; the filtrate is waiting for determination.

5.5 Instrument reference conditions

- a) Chromatographic column: C18 column (column-length 250mm, inner-diameter 4.6mm, particle-size 5 μ m), or the chromatographic column with equivalent performance.
- b) Mobile phase: methanol-phosphate buffer solution (70+30, volume ratio).
- c) Flow rate: 1.0mL/min.
- d) Injection volume: 10 μ L.
- e) Column temperature: room temperature.
- f) Fluorescence detector: excitation wavelength 390nm, emission wavelength 480nm.

5.6 Drawing of standard working curve

Respectively PIPETTE the above standard series of derivatization solution into liquid chromatograph; DETERMINE the corresponding peak area according to 5.5 instrument reference conditions (see Appendix A for the chromatogram of 11-amino undecanoic acid standard substance derivative). With the 11-amino undecanoic acid concentration of standard working solution as abscissa, AND the corresponding peak area of 11-amino undecanoic acid as vertical axis, DRAW a standard working curve.

5.7 Determination of sample solution

According to 5.5 instrument reference conditions, INJECT the sample derivative solution into liquid chromatograph; DETERMINE the nature based on the retention time; at the same time, RECORD peak area; OBTAIN the concentration of 11-amino undecanoic acid in the solution-under-test according to the standard curve.

6 Analysis results expression

OBTAIN 11-amino undecanoic acid concentration of food stimulant sample solution from standard curve; CALCULATE migration quantity according to GB