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National food safety standard – Food contact materials and articles - Determination of migration of 1-octene and tetrahydrofuran

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Table of Contents

Foreword

This Standard replaces GB/T 23296.4-2009, *Food contact materials - Polymer - Determination of 1-octene and tetrahydrofuran in food simulants - Gas chromatography.*

Compared with GB/T 23296.4-2009, the major changes of this Standard are as follows:

-- the standard name is changed into "*National food safety standard - Food contact materials and articles - Determination of migration of 1-octene and tetrahydrofuran*";

-- the gas chromatography-mass spectrometry is added;

-- the preparation of food simulant test solutions is changed;

-- the expression of analysis results is changed;

-- the detection limits and quantitation limits are changed.

National food safety standard – Food contact materials and articles - Determination of migration of 1-octene and tetrahydrofuran

1 Scope

This Standard specifies the method for the determination of migration of 1-octene and tetrahydrofuran in food contact materials and articles.

This Standard applies to the determination of migration of 1-octene and tetrahydrofuran in food contact materials and articles.

Method I -- Gas chromatography

2 Principle

1-octene and tetrahydrofuran in food simulants are separated from internal standard substances and other components in a chromatographic column after headspace sample injection; a hydrogen flame ionization detector is used for testing; and the internal standard method is used for quantitation.

3 Reagents and materials

Unless indicated otherwise, all reagents used for this method are analytically pure and the water is grade one water specified in GB/T 6682.

3.1 Reagents

3.1.1 *N,N*-Dimethylacetamide (DMAC, C_4H_9NO): of purity > 99.0%.

3.1.2 Water-based, acidic, alcoholic and oil-based food simulants: the reagents used are as specified in GB 31604.1.

3.2 Reagent preparation

3.2.1 Water-based, acidic, alcoholic and oil-based food simulants: the operation is as specified in GB 5009.156.

3.3 Standard substances

3.4.6 Isooctane internal standard intermediate solution (120 μg/mL): transfer 3.0 mL of isooctane internal standard stock solution to a 50 mL volumetric flask and use DMAC to add to scale. The storage conditions are the same as those specified in 3.4.1.

3.4.7 Water-based, acidic and alcoholic THF standard stock solution (2.2 mg/mL): measure 99 mL of water to pour into a 100 mL volumetric flask and weigh with the stopper (accurate to 0.000 1 g). Add 220 mg of THF, about 250 μL; weigh once again (accurate to 0.000 1 g); and use water to add to scale. Calculate the concentration of THF accurately. Store in a dark place at 4°C, with a period of validity of one week.

3.4.8 Water-based, acidic and alcoholic THF standard intermediate solution (22 μg/mL): measure 90 mL of water to pour into a 100 mL volumetric flask; transfer 1.0 mL of water-based THF standard stock solution; and use water to add to scale. The storage conditions are the same as those specified in 3.4.7.

3.4.9 Water-based, acidic based and alcoholic food simulant medium standard working solution of THF: measure respectively 45 mL of water-based, acidic and alcoholic food simulants to pour into six 50 mL volumetric flasks. Add a certain volume of water-based, acidic and alcoholic THF standard intermediate solution in accordance with Table A.2 and use corresponding food simulants to add to scale.

Accurately transfer 10.0 mL of the above-mentioned solutions to six headspace bottles respectively and immediately seal using insulator and aluminium cover. Use a micro-syringe to transfer 100 μL of water-based, acidic and alcoholic THP internal standard intermediate solution to all headspace bottles through the insulator, and mix up.

3.4.10 Water-based, acidic based and alcoholic THP internal standard stock solution (1 mg/mL): weigh 99 mL of water to pour into a 100 mL volumetric flask; weigh with the stopper (accurate to 0.000 1 g); add 100 mg of THP, about 150 μL; weigh once again (accurate to 0.000 1 g); and use water to add to scale. Accurately calculate the concentration of THP. The storage conditions are the same as those specified in 3.4.7.

3.4.11 Water-based, acidic based and alcoholic THP internal standard intermediate solution (70 μg/mL): measure 90 mL of water to pour into a 100 mL volumetric flask; transfer 7.0 mL of water-based THP internal standard stock solution; and use water to add to scale. The storage conditions are the same as those specified in 3.4.7.

3.4.12 Oil-based THF standard stock solution (1.0 mg/mL): measure 45 mL of DMAC to pour into a 50 mL volumetric flask and weigh with the stopper (accurate to 0.000 1 g). Then add 50 mg of THF, about 60 μL; weigh once again (accurate to 0.000 1 g); and use DMAC to add to scale. Accurately calculate the concentration of THF. The storage conditions are the same as those specified in 3.4.7.

3.4.13 Oil-based THF standard intermediate solution (50 μg/mL): transfer 1.0 mL of oil-based THF standard stock solution to a 20 mL volumetric flask and use DMAC to add to scale. The storage conditions are the same as those specified in 3.4.7.

In accordance with the requirements of GB 5009.156 and GB 31604.1, carry out migration test for sample and obtain food simulant test solutions. If a food simulant test solution obtained is not ready for further test at once, it shall be stored in a refrigerator at 0°C~4°C, which shall be kept out of the sun.

Cool or restore the food simulant test solutions to room temperature for further test.

5.2 Test solution preparation

5.2.1 Preparation of water-based, acidic and alcoholic food simulant test solutions for the determination of 1-octylene

Transfer 1.0 mL of water-based food simulant from the migration test to a headspace bottle, and immediately seal using insulator and aluminium cover. Use a micro-syringe to add in turn 200 μL of isooctane internal standard intermediate solution and 200 μL of DMAC through the insulator; and mix up.

5.2.2 Preparation of olive oil medium food simulant test solution for the determination of 1-octylene

Weigh 1 g (accurate to 0.01 g) of olive oil medium food simulant from the migration test to place in a headspace bottle; and hereafter, process in accordance with the operating procedures of 5.2.1 “and immediately seal using insulator and aluminium cover …”.

5.2.3 Preparation of water-based, acidic and alcoholic food simulant test solution for the determination of tetrahydrofuran

Transfer 10.0 mL of water-based, acidic and alcoholic food simulant test solution from the migration test to a headspace bottle, and immediately seal using insulator and aluminium cover. Use a micro-syringe to add 100 μL of water-based, acidic and alcoholic THP internal standard intermediate solution through the insulator; and mix up.

5.2.4 Preparation of olive oil medium food simulant test solutions for the determination of tetrahydrofuran

Weigh 10 g (accurate to 0.01 g) of olive oil medium food simulant from the migration test to place into a headspace bottle, and immediately seal using insulator and aluminium cover. Use a micro-syringe to add 100 μL of oil-based THP internal standard intermediate solution (3.4.16) through the insulator; then use a micro-syringe to add rapidly 300 μL of DMAC; and mix up.

5.3 Preparation of blank test solutions

Process the food simulants in accordance with the procedures specified in 5.2, which are not in contact with food contact materials.

5.4 Determination

f) sampling method: split stream sampling with a split ratio of 10:1;

g) sample volume: 250 μL;

h) transmission line temperature: 250°C;

i) ionization mode: EI;

j) solvent delay: 2.2 min;

k) mass spectrum scanning method: selected ion mode (SIM), turning off filament after 8 min;

l) isooctane: quantitative ion *m/z* 57, qualitative ions *m/z* 58 and 99; 1-octene: quantitative ion *m/z* 70, qualitative ions *m/z* 83 and 112; tetrahydrofuran: quantitative ion *m/z* 72, qualitative ions *m/z* 71 and 73; tetrahydropyrane: quantitative ion *m/z* 85, qualitative ions *m/z* 67 and 86.

12.4.2 Plotting of standard working curves

Carry out determination of standard working solutions in turn in accordance with the determination conditions listed in 12.4.1. Plot standard working curves using the 1-octene or tetrahydrofuran concentrations in standard working solutions as the abscissa, in mg/L or mg/kg, and the peak area ratio of 1-octene/isooctane or tetrahydrofuran/tetrahydropyrane as the ordinate. For the total ion current chromatogram of gas chromatography-mass spectrometry refer to Annex B.

12.4.3 Confirmation

Take test solutions (12.2) to carry out the confirmatory test of the internal standard method in accordance with the gas chromatographic-mass spectrometric conditions described in 12.4.1. Refer to the gas chromatographic-mass spectrometric total ion current chromatogram of 1-octene or tetrahydrofuran in Figure B.3 and Figure B.4. Under the same test conditions, if the retention time of chromatographic peak is within ± 2.5% of that of standard solution; and if after deducting background, the relative abundance of qualitative ions in the mass chromatogram of sample compares with the relative abundance of qualitative ions corresponding to the chromatogram of standard solution of similar concentration, the relative abundance deviations not exceeding those specified in Table 1, then it can be determined that substances to be tested exist in sample.

Table 1 -- Maximum allowable deviation of relative ion abundance during qualitative confirmation

Relative ion abundance	> 50%	20%~50%	10%~20%	≤ 10%
Allowable relative deviation	± 20%	± 25%	± 30%	± 50%

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