GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

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National Food Safety Standard – Food Contact Materials and Articles – Determination of the Content and Migration of Phthalate Esters

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National Food Safety Standard – Food Contact Materials and Articles – Determination of the Content and Migration of Phthalate Esters

1 Scope

This Standard specifies the determination of the content and migration of phthalate esters in the food contact materials and articles.

This Standard is applicable to the determination of the content of phthalate esters in the food plastic packaging materials, and the determination of migration of phthalate esters in food contact materials and articles.

Determination of Phthalate Esters

2 Principle

After crushing the food plastic packaging materials and articles, use n-hexane to conduct the ultrasonic extraction; after filtering the extracting solution, use gas chromatography-mass spectrometry to determine. Adopt the feature selective ion monitoring scan mode (SIM), so that retain the qualitative of abundance ratio of the time and fragments; use external standard method to quantify.

3 Reagents and Materials

Unless otherwise is specified, the reagents used in this Method are all chromatographic pure (or analytically pure that has been redistilled). Water shall be the one redistilled by a full glass device and stored in the glass vessel.

3.1 Reagents

3.1.1 N-hexane (C_6H_{14}).

3.12 Acetone (CH_3COCH_3).

3.2 Standard substance of phthalate esters

5 Analytical Procedures

5.1 Specimen preparation

Take 5g of typical sample; shear the specimen (excluding the preservative film) into single pieces with diameter≤0.2cm; the preservative film piece diameter≤0.3cm; then mix evenly; accurately take 0.2g~0.5g of specimen (accurate to 0.0001g) into stoppered triangular flask; add 20mL of n-hexane; perform ultrasonic extraction for 30min and filter; after than the residue is re-extracted once by 20mL of n-hexane; combine the filtrate into 50mL volumetric flask; use n-hexane to make constant volume to the scale; then dilute it as per the content of phthalate esters in the specimen; after mixing evenly, pass through a 0.45μm organic phase glass filter film; and be analyzed in the gas chromatograph-mass spectrometer.

5.2 Blank test

The reagents used in the test shall be treated as per 5.1; and analyzed in GC-MS.

5.3 Instrument reference conditions

5.3.1 Gas chromatography reference conditions

a) Chromatographic column: 5% polyphenylmethylsiloxane quartz capillary column or analytical column with similar performance; the specification indicates column length of 30.0m, inner diameter of 0.25mm, film thickness of 0.25μm;

b) Sample inlet temperature: 260°C;

c) Temperature rise program: maintain the initial column temperature of 60°C for 1min; raise temperature to 220°C by 20°C/min, maintain for 1min; raise temperature to 250°C by 5°C/min, maintain for 1min; then raise temperature to 290°C by 20°C/min, maintain for 7.5min;

d) Carrier gas: helium (purity >99.999%), flow rate: 1mL/min;

e) Sampling injecting method: splitless sample injection;

f) Sample injecting volume: 1μL.

5.3.2 Mass spectrometry reference conditions

a) Gas chromatography and mass spectrometry interface temperature: 280°C;

b) Ion source temperature: 230°C;

c) Ionization mode: electron impact ionization source (EI);

10 Reagents and Materials

Unless otherwise is specified, the reagents used in this Method are all chromatographic pure (or analytically pure that has been redistilled). Water shall be the one redistilled by a full glass device and stored in the glass vessel.

10.1 Reagents

10.1.1 N-hexane (C_6H_{14}).

10.1.2 Absolute alcohol (C_2H_5OH).

10.1.3 Acetic acid (CH_3COOH).

10.1.4 Reagents required for preparing the acidic and alcoholic food simulants: abide by the provisions of GB 31604.1.

10.1.5 Isooctane: oil-based food simulants.

10.2 Standard substance

The standard substance for 18 kinds of phthalate esters, the same as 3.2.

10.3 Preparation of standard solution

10.3.1 Standard stock solution: the same as 3.3.1.

10.3.2 17 kinds of phthalate esters standard use solutions: the same as 3.3.2.

10.3.3 Diisononyl ortho-phthalate standard use solution: the same as 3.3.3.

10.3.4 Phthalate ester standard serial working solution: accurately pipette appropriate amount of phthalate ester standard use solution; use n-hexane to prepare 17 kinds of phthalate ester mixing standard use solutions with concentrations of 0.1mg/L, 0.2mg/L, 0.5mg/L, 1.0mg/L, 3.0mg/L, 8.0mg/L, 10.0mg/L, as well as single-labeled diisononyl ortho-phthalate (DINP) with concentrations of 0.5mg/L, 1.0mg/L, 3.0mg/L, 8.0mg/L, 10.0mg/L for later-use.

11 Apparatus

11.1 Gas chromatography-mass spectrometer: with electron impact source (EI).

11.2 Centrifuge.

11.3 Oscillator.

11.4 Solid phase extraction device.

11.5 Constant temperature oven.

11.6 Low-temperature incubator.

11.7 Centrifuge tube: 10mL.

11.8 Volumetric flask: 10mL.

11.9 Glassware.

NOTE: after washing the used glassware, rinse by the distilled water for 3 times; soak in acetone for 1h; bake at 200°C for 2h; cool off to the room temperature for later-use.

12 Analytical Procedures

12.1 Preparation of food simulant test solution

According to the requirements of GB 5009.156 and GB 31604.1, perform the migration test against the sample; then obtain the food simulant test solution.

12.2 Treatment of soaking solution

Place the soaking solution at the room temperature; for the isooctane and 95% ethanol aqueous solution (≥ 50% alcoholic food) simulant soaking solution, accurately take 10g±0.01g of soaking solution into the pear-shaped distillation flask; reduce pressure in 45°C water bath; perform rotary evaporation to dryness; add 1mL of n-hexane to dissolve; perform vortex oscillation for 3min; centrifuge for 5min at 4000r/min; collect the supernatant for the instrument test.

For other simulant soaking solutions (including water-based, acidic food, <50% alcoholic food simulant), take 10g±0.01g of simulant soaking solution into 25mL centrifuge tube; add 4mL of n-hexane; shake and extract for 10min; centrifuge for 5min at 4000r/min speed; take out the upper layer of n-hexane; repeatedly extract twice; combine the n-hexane phase into pear-shaped distillation flask; reduce pressure in 45°C water bath; perform rotary evaporation to dryness; add 1mL of n-hexane to dissolve; perform vortex oscillation for 3min; centrifuge for 5min at 4000r/min; collect the supernatant for the instrument test.

The sample solution can be diluted as per the specific conditions, so that the measured value shall be within the linear range of the standard curve.

12.3 Blank test

The reagents used in the test shall be treated as per 12.2; perform the GC-MS analysis.

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