GB

NATIONAL STANDARD OF THE

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National Food Safety Standard – Determination of Solvent Residues in Food Contact Materials and Products

食品安全国家标准 食品接触材料及制品 溶剂残留量的测定

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National Food Standard – Determination of Solvent Residues in Food Contact Materials and Products

1 Scope

This Standard specifies the gas chromatography and gas chromatography-mass spectrometry to determine the residues of 25 solvents, such as cyclohexane, methylcyclohexane, acetone, ethyl acetate, methanol, isopropyl acetate, butanone, isopropyl alcohol, ethanol, n-propyl acetate, 4-Methyl-2-pentanone, n-propanol, butyl acetate, isobutanol, n-butanol, ethylene glycol methyl ether acetate, propylene glycol methyl ether acetate, propylene glycol methyl ether, propylene glycol ethyl ether, benzene, toluene, ethylbenzene, o-xylene, p-xylene, and m-xylene in the food contact composite materials and products.

This Standard is applicable to the determination of solvent residues in food contact composite materials and products.

Method-I Gas Chromatography

2 Principle

Food contact materials and product samples are placed in headspace bottles and heated to make the components to be tested reach liquid-gas equilibrium. After the headspace gas is separated by a gas chromatography column, a hydrogen flame ionization detector is used for detection by the external standard method Quantitative.

3 Reagents and Materials

3.1 Reagents

N, *N*-dimethylformamide (C_3H_7NO): chromatographically pure.

3.2 Standard products

Cyclohexane, methylcyclohexane, acetone, ethyl acetate, methanol, isopropyl acetate, butanone, isopropyl alcohol, ethanol, n-propyl acetate, 4-Methyl-2-pentanone, n-propanol, butyl acetate, isobutanol, n-butanol, ethylene glycol methyl ether acetate, propylene glycol methyl ether acetate, propylene glycol methyl ether, propylene glycol ethyl ether, benzene, toluene, ethylbenzene, o-xylene, p-xylene: purity ≥98%, or standard substances certified by the

state and awarded a standard substance certificate. For detailed information, see Table A.1 of Appendix A.

3.3 Preparation of standard solution

3.3.1 Single standard substance stock solution (200g/L): Accurately weigh 2g (accurate to 0.1mg) of single standard product; dissolve it with *N*, *N*-dimethylformamide and make constant volume to 10mL; mix well; and transfer the solution to a brown glass container; and store in the dark at -18°C. It shall be kept valid for 6 months.

3.3.2 Non-benzene solvent mixed standard intermediate solution (10g/L): Respectively pipette 2.50mL of each non-benzene solvent single standard substance stock solution (200g/L) into a 50mL volumetric flask; add *N*, *N*-dimethylformamide to make constant volume; mix well; transfer the solution to a brown glass container and store it in the dark at -18°C. It shall be kept valid for 6 months.

3.3.3 Benzene solvent mixed standard intermediate solution (10g/L): Respectively pipette 2.50mL of each benzene solvent single standard substance stock solution (200g/L) into a 50mL volumetric flask, and add *N*, *N*-dimethylformamide to make constant volume; mix well; transfer the solution to a brown glass container and store it in the dark at -18°C. It shall be kept valid for 6 months.

3.3.4 Mixed standard intermediate solution (non-benzene solvent 5000mg/L, benzene solvent 2500mg/L): Respectively pipette 25.00mL of non-benzene mixed standard intermediate solution (10g/L) and 12.50mL of benzene mixed standard intermediate solution (10g/L) in a 50mL volumetric flask; add *N*, *N*-dimethylformamide to make constant volume; mix well; and transfer the solution to a brown glass container. The mass concentration of each non-benzene solvent in the obtained mixed standard intermediate solution is 5000 mg/L; and the mass concentration of the benzene solvent is 2500 mg/L. Store it in the dark at -18°C; it shall be kept valid for 6 months.

3.3.5 Mixed standard series intermediate solution: Respectively pipette 0.20mL, 0.40mL, 1.0mL, 2.0mL, 5.0mL, 10.0mL of the mixed standard intermediate solution (non-benzene solvent 5000mg/L, benzene solvent 2500mg/L) into six 10mL volumetric flasks; add *N*, *N*-dimethylformamide to make constant volume; and mix evenly; and obtain six mixed standard series intermediate solutions. The mass concentration of each non-benzene solvent in the 6 mixed standard series intermediate solutions is 100mg/L, 200mg/L, 500mg/L, 1000mg/L, 2500mg/L, 5000mg/L; and the mass concentration of each benzene solvent is 50mg/L, 100mg/L, 250mg/L, 500mg/L, 1250mg/L and 2500mg/L in sequence; it shall be prepared for immediate use.

3.3.6 Mixed standard series working solution: Use a micro-injection needle to accurately pipette 10μL of the above 6 mixed standard series intermediate solutions into 6 headspace bottles containing blank matrix; and quickly cap them. The masses of each non-benzene solvent in the obtained system to be tested are 1μg, 2μg, 5μg, 10μg, 25μg, and 50μg respectively; and the

N, *N*-dimethylformamide; and seal it for testing. When the target solvent residual amount in the specimen exceeds the range of the standard curve, the sampling area can be appropriately reduced.

5.2.3 Other composite packaging materials and products

Cut a specimen of 100cm^2 (accurate to 0.01cm^2 based on one side); quickly cut the specimen into long strips of about 1cm×3cm; place it in a headspace bottle as soon as possible. Add 10μL of *N*, *N*-dimethylformamide; seal it for testing. When the target solvent residual amount in the specimen exceeds the range of the standard curve or the volume occupied by the specimen exceeds 2/3 of the volume of the headspace bottle; the sampling area can be appropriately reduced.

5.2.4 Preparation of blank matrix

Except that *N*, *N*-dimethylformamide is not added, prepare the specimen according to 5.2.1~5.2.3. Prepare at least 7 parallel samples; place them in open headspace bottles respectively; then place them in a vacuum drying oven for vacuum drying at 90°C for 24 h. If the target solvent is still detected in the blank matrix after the first vacuum drying, perform a second vacuum drying until no target solvent is detected in the blank matrix. The blank matrix shall be stored in a desiccator for later use.

5.3 Blank test

Take the blank matrix prepared in 5.2.4, add 10μL of *N*, *N*-dimethylformamide, and seal it for testing.

5.4 Instrument reference conditions

5.4.1 Headspace sampler reference conditions

The reference conditions for the headspace sampler are as follows:

a) Equilibrium temperature: 80℃;

b) Injection loop temperature: 90℃;

c) Transmission line temperature: 100℃;

d) Headspace equilibration time: 30min;

e) Ring equilibrium time: 0.05min;

f) Pressurization time: 0.2min;

g) Injection time: 0.5min.

5.4.2 Gas chromatography reference conditions

The gas chromatography reference conditions are as follows:

a) Chromatographic column: Polyethylene glycol capillary column, length 50m, inner diameter 0.25mm, film thickness 0.2μm or equivalent performance;

b) Heating program: initial temperature is maintained at 35°C for 10 min; heated to 100°C at a heating rate of 3°C/min; then raised to 130°C at a rate of 10°C/min, maintained for 5 min; and then run for 2 min;

c) Inlet temperature: 230℃;

d) Split ratio: 2:1;

e) Septum purge: 10mL/min;

f) Detector temperature: 280℃;

g) The flow rates of hydrogen, air, and makeup gas are: 30mL/min, 300mL/min, and 25mL/min, respectively;

h) Flow rate: maintain 0.5mL/min for 10min; increase to 1mL/min at a constant speed for 0.5min; and maintain 1mL/min until the end of the programmed temperature rise.

5.5 Drawing of standard curve

According to the instrument reference conditions in 5.4, measure the mixed standard series working solution. Taking the mass of each target solvent in the mixed standard working solution as the abscissa and the corresponding peak area as the ordinate, draw the standard curve of each target solvent to obtain linear equation. For the chromatogram of the mixed standard working solution, see Figure B.1 of Appendix B.

5.6 Determination of sample solution

According to the instrument reference conditions in 5.4, measure the sample (5.2) and blank test sample (5.3), and calculate the mass of each target solvent from the standard curve.

6 Expression of Analysis Results

The residual amount of each solvent in food contact materials and products is calculated according to Formula (1).

$$X = \left(\frac{m}{S} - \frac{m_0}{S_0}\right) \times 10 \qquad \cdots\cdots(1)$$

Where:

The same as 3.2.

10.3 Preparation of standard solution

10.3.1 Single standard material stock solution (200g/L): The same as 3.3.1.

10.3.2 Non-benzene solvent mixed standard intermediate solution (10g/L): The same as 3.3.2.

10.3.3 Benzene solvent mixed standard intermediate solution (10g/L): The same as 3.3.3.

10.3.4 Mixed standard intermediate solution (non-benzene solvent 5000mg/L, benzene solvent 500mg/L): Respectively pipette 25.0mL of non-benzene solvent mixed standard intermediate solution (10g/L) and 2.5mL of benzene solvent mixed standard intermediate solution (10g/L) into a 50mL volumetric flask; add *N*, *N*-dimethylformamide to make the constant volume; mix well; and transfer the solution to a brown glass container. The mass concentration of non-benzene solvents in the obtained mixed standard intermediate solution is all 5000 mg/L; and the mass concentration of benzene solvents is 500 mg/L. Store at -18°C away from light, valid for 6 months.

10.3.5 Mixed standard series intermediate solution: Respectively pipette 0.20mL, 0.40mL, 1.0mL, 2.0mL, 5.0mL, 10.0mL of mixed standard intermediate solution (non-benzene solvent 5000mg/L, benzene solvent 500mg/L) into six 10mL volumetric flasks; add *N*, *N*-dimethylformamide to make the constant volume; mix evenly to obtain six mixed standard intermediate solutions. The mass concentration of each non-benzene solvent in the 6 mixed standard intermediate solutions is 100mg/L, 200mg/L, 500mg/L, 1000mg/L, 2500mg/L, 5000mg/L; and the mass concentration of each benzene solvent is 10mg/L, 20mg/L, 50mg/L, 100mg/L, 250mg/L, 500mg/L; and it is prepared for immediate use.

10.3.6 Mixed standard series working solution: Use a micro-injection needle to accurately transfer 10μL of the above 6 mixed standard series intermediate solutions into 6 headspace bottles containing blank matrix; and quickly cap them. The masses of each non-benzene solvent in the obtained system to be tested are 1μg, 2μg, 5μg, 10μg, 25μg, and 50μg, respectively; and the masses of each benzene solvent are 0.1μg, 0.2μg, 0.5μg, 1μg, 2.5μg, and 5μg, respectively. It shall be prepared for immediate use.

11 Instruments and Equipment

11.1 Gas chromatograph-mass spectrometer: Equipped with headspace sampler and electron impact source EI.

11.2 The same as 4.2.

11.3 The same as 4.3.

11.4 The same as 4.4.

11.5 The same as 4.5.

11.6 The same as 4.6.

12 Analysis Procedures

12.1 Sample storage

The same as 5.1.

12.2 Preparation of specimen

The same as 5.2.

12.3 Blank test

The same as 5.3.

12.4 Instrument reference conditions

12.4.1 Headspace sampler reference conditions

The reference conditions for the headspace sampler are as follows:

a) Equilibrium temperature: 80℃;

b) Injection loop temperature: 90℃;

c) Transmission line temperature: 100℃;

d) Headspace equilibration time: 30min;

e) Ring equilibrium time: 0.05min;

f) Pressurization time: 0.2min;

g) Injection time: 0.5min.

12.4.2 Gas chromatography-mass spectrometry reference conditions

The gas chromatography reference conditions are as follows:

a) Chromatographic column: Polyethylene glycol capillary column, length 30m, inner diameter 0.32mm, film thickness 0.25μm or equivalent performance;

b) Temperature rising program: Initial temperature is 50 ℃ and maintained for 3 min; heated to 70 ℃ at a heating rate of 10 ℃/min and maintained for 1 min; heated to 160 ℃ at a

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