



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

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**National food safety standard - Determination of solvent
residues in food contact materials and products**

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Table of Contents

1 Scope	3
2 Principle	3
3 Reagents and Materials	3
4 Instruments and Equipment	5
5 Analysis Procedures	5
6 Expression of Analysis Results	7
7 Precision	8
8 Others	8
9 Principle	8
10 Reagents and Materials	8
11 Instruments and Equipment	9
12 Analysis Procedures	10
13 Expression of Analysis Results	12
14 Precision	12
15 Others	12
Appendix A Basic Information of Standard Product of Solvent	13
Appendix B Mixed standard working solution chromatogram	14
Appendix C Solvent Quantitative and Qualitative Ion List	16

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.

3.1 Reagents

N, N-dimethylformamide (C₃H₇NO): 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 $\geq 98\%$, or standard substances certified by the state and awarded a standard substance certificate. For detailed information, see Table A.1 of

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

10uL 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 1ug, 2ug, 5ug, 10ug, 25ug, and 50ug 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 100cm² (accurate to 0.01cm² based on one side); quickly cut the specimen into long strips of about 1cmx3cm; place it in a headspace bottle as soon as possible. Add 10uL 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 10 μ L 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 degrees C;
- b) Injection loop temperature: 90 degrees C;
- c) Transmission line temperature: 100 degrees C;
- 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 0.2 μ m 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 degrees C;
- d) Split ratio: 2:1;
- e) Septum purge: 10mL/min;
- f) Detector temperature: 280 degrees C;
- 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