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National Food Safety Standard - Food Contact Materials and Articles - Determination of Diaminomethylbezen in Complex Food Contact Materials and Articles

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National Food Safety Standard - Food Contact Materials and Articles - Determination of Diaminomethylbezen in Complex Food Contact Materials and Articles

1 Scope

This Standard specifies the method for determination of diaminomethylbezen of complex for food packaging material.

This Standard applies to the determination of diaminomethylbezen of complex for food packaging material.

Method I Gas chromatography

2 Principles

USE 4% acetic acid solution to leach the diaminomethylbezen in the sample; after the leaching solution is cooled and extracted with dichloromethane under alkaline conditions, ADD heptafluorobutyric anhydride to derivatize; then, INJECT the derivatives into a gas chromatograph with electron capture detector for determination; USE retention time to determine the quality, and the external standard method to quantify.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method are analytically pure. The water is the Grade III water specified in GB/T 6682.

3.1 Reagents

3.1.1 Dichloromethane (CH_2Cl_2): chromatographically pure.

3.1.2 Tert-butyl methyl ether ($C_5H_{12}O$): chromatographically pure.

3.1.3 Heptafluorobutyric anhydride ($C_8F_{14}O_3$): chromatographically pure.

3.1.4 Anhydrous sodium sulfate (Na_2SO_4).

5 Analytical procedures

5.1 Preparation of samples

5.1.1 Packaging material which does not contain food ever: USE water to wash for 3 times; DRY it; at 2 mL/cm^2, FILL it with acetic acid solution; and heat seal.

5.1.2 Packaging material which contains food ever: SNIP the seal; REMOVE all the food; USE clean water to rinse until there is no dirt; and USE water to wash for 3 times; after drying it, at 2 mL/cm^2, FILL it with acetic acid solution; and heat seal.

5.2 Migration test

PUT the above 5.1.1 or 5.1.2 heat-sealed packaging material (The operating temperature is 60 °C~120 °C) in an oven pre-adjusted to 120 °C±5 °C; KEEP it at constant temperature for 40 min; TAKE it out and COOL naturally to room temperature; then SNIP the seal; and REMOVE the extract into a dry beaker; SET aside.

PUT the packaging material (The operating temperature is less than 60 °C) in an oven pre-adjusted to 60 °C±5 °C; KEEP it at constant temperature for 2 h; TAKE it out and COOL naturally to room temperature; then SNIP the seal; and REMOVE the water into a dry beaker; SET aside.

5.3 Derivatization process

MEASURE 50.0 mL of the sample; PLACE it in a separatory funnel; USE sodium hydroxide solution to adjust the pH to 8.0; MIX well; and ADD 10 g of sodium chloride and MIX well; then USE 10 mL of dichloromethane to extract twice, 5 min each time; LET it stand for 10 min. COMBINE the two extracts; after being dehydrated with anhydrous sodium sulfate, at 40°C, BLOW nitrogen until nearly dry; ADD 2 mL of dichloromethane and MIX well; and ADD 100 μL of heptafluorobutyric anhydride, MIX gently; at room temperature, CARRY out derivatization reaction for 15 min.

TRANSFER the above reaction solution into a 60 mL separatory funnel; USE 2 mL of dichloromethane to wash the concentration flask several times; INCORPORATE the washing liquor into the separatory funnel; ADD 5 mL of sodium bicarbonate solution; gently SHAKE it for 2 min; LET it stand for 5 min; TRANSFER the dichloromethane layer into a 10 mL test tube; at 40°C, BLOW nitrogen until nearly dry; USE tert-butyl methyl ether to dissolve and dilute to 5.00 mL; and INJECT it into gas chromatograph for analysis.

5.4 Preparation of standard determination solution of

10 Reagents and materials

Same as those under 3.

11 Instruments and equipment

11.1 Gas chromatography-mass spectrometry.

11.2 Analytical balance: The sensitivity is 0.01 mg and 0.01 g.

11.3 Nitrogen blowing concentrator.

11.4 pH meter: The precision is 0.1.

12 Analytical procedures

12.1 Preparation of samples

Same as that under 5.1.

12.2 Migration test

Same as that under 5.2.

12.3 Derivatization process

Same as that under 5.3.

12.4 Preparation of standard determination solution of diaminomethylbezen

Same as that under 5.4.

12.5 Reference conditions for gas chromatography-mass spectrometry

a) Chromatographic column: HP-5 MS. The column length is 30 m. The inner diameter is 0.25 mm. The film thickness is 0.25 μm. Or chromatographic column with equivalent performance;

b) Column temperature program: The initial temperature is 60 °C and maintained for 2 min. At 15 °C/min, the temperature is raised to 240 °C and maintained for 5 min;

c) Inlet temperature: 200 °C;

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