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Determination of Polybrominated Biphenyls and Polybrominated Diphenyl Ethers in Plastic Products - High Performance Liquid Chromatography

塑料制品中多溴联苯和多溴二苯醚的测定

高效液相色谱法

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Determination of Polybrominated Biphenyls and Polybrominated Diphenyl Ethers in Plastic Products - High Performance Liquid Chromatography

1 Scope

This Standard stipulates the high performance liquid chromatography for polybrominated biphenyls (abbreviation: PBBs; please refer to Figure 1 for its structural formula) and polybrominated diphenyl ethers (abbreviation: PBDEs; please refer to Figure 2 for its structural formula).

This Standard is applicable to the determination of polybrominated biphenyls and polybrominated diphenyl ethers in plastic products.

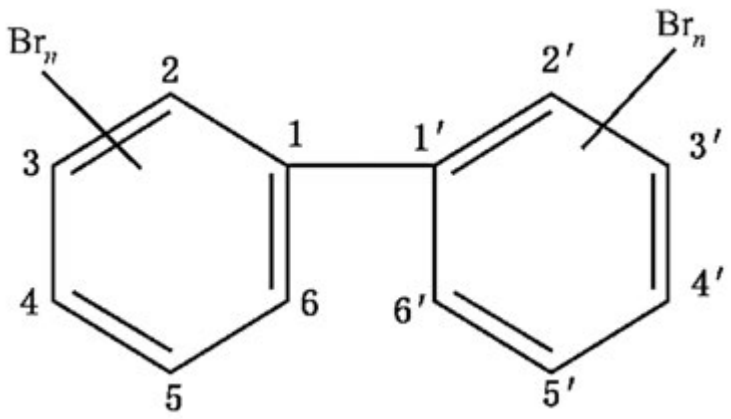

Figure 1 -- Structure of Polybrominated Biphenyls

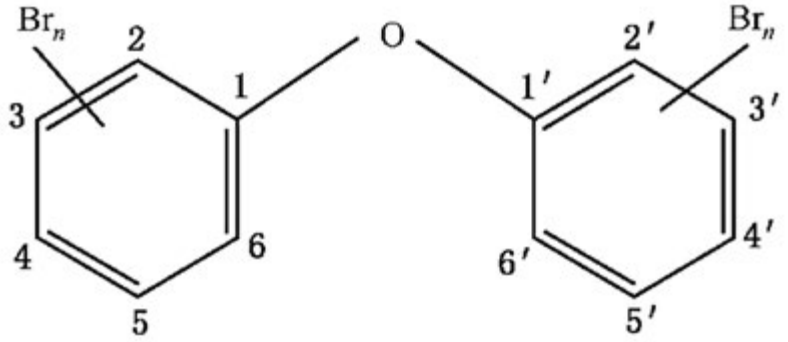

Figure 2 -- Structure of Polybrominated Diphenyl Ethers

4.4 Rotary evaporator.

4.5 Pulverizer.

4.6 Solid-phase extraction device.

4.7 Constant-temperature water bath (temperature accuracy shall be less than 1 °C).

4.8 Analytical balance (accurate to: 0.0001 g).

4.9 Centrifuge.

4.10 Closed microwave extractor.

4.11 Organic-phase filtration membrane: 0.45 μm filtration membrane.

5 Sample Preparation

In accordance with product's property, through the modes of clipping, crushing or liquid nitrogen freezing and pulverization, prepare sample into particles with the particle size of less than 1 mm.

6 Analytical Procedures

6.1 Extraction

Extraction includes the following three methods:

a) Soxhlet extraction method: on the analytical balance, accurately weigh-take 0.1 g of sample (accurate to 1 mg); place it into a filtration paper cylinder. Add 150 mL of dichloromethane solution, then, control the water bath temperature at 60 °C ± 5 °C. At the rate of 2 drops/s ~ 3 drops/s, conduct extraction for over 12 h. After the extraction is completed, in terms of relatively clean sample or slightly turbid sample, which can be improved through the adding of methanol, use the rotary evaporator to concentrate the extract and reach a constant volume of 100 mL. Then, use 0.45 μm filtration membrane (see 4.11) to filter it; directly analyze it. In terms of dark-colored sample with a lot of impurities, use the rotary evaporator to concentrate the extract to a smaller volume, then, conduct purification treatment in accordance with 6.2.

b) Microwave extraction method: accurately weigh-take 0.5 g ~ 2 g of sample, which is already pulverized, accurate to 1 mg. Place it into an extraction tank, then, accurately transfer-take 20 mL of methylbenzene-methanol (see 3.7); seal it up and place it in the microwave extractor (see 4.10). Within 5 min, raise the temperature to 115 °C, maintain for over 15 min, then, cool it down to the room temperature. Completely transfer the extract; use the extracting solvent

e) Flow rate: 1.0 mL/min;

f) Injection volume: 20 μL.

6.3.2 Liquid chromatographic determination

In accordance with the PBBs and PBDEs content being tested in the sample solution, select a standard solution (see 3.6) with an approximate concentration, conduct iso-volumetric interspersed sample injection. The response value of PBBs and PBDEs in the standard solution and the sample solution to be tested shall be within the linear range of the standard solution. In accordance with the chromatographic peak area, use external standard method to quantify it.

Please refer to Figure A.1 in Appendix 1 and Figure B.1 in Appendix B for the typical chromatogram of some PBBs and PBDEs standard substances.

6.3.3 Blank test

Except from the step of adding sample, comply with the other above-mentioned steps for determination.

7 Result Calculation

In accordance with Formula (1), calculate the mass fraction of PBB or PBDE in the sample:

$$w = [(A_i - A_0) \cdot \rho \cdot V] / (A \cdot m) \qquad \cdots\cdots (1)$$

Where,

w---mass fraction of PBBs/PBDEs in the sample, expressed in (mg/kg);

A_i---chromatographic peak area of PBBs and PBDEs in the sample solution;

A_0---chromatographic peak area in the blank sample;

A---chromatographic peak area of PBBs and PBDEs in the standard working solution;

ρ---mass concentration of PBBs and PBDEs in the standard working solution, expressed in (mg/L);

V---final constant volume of the sample solution, expressed in (mL);

m---sample mass represented by the final sample solution, expressed in (g).

The calculation result shall retain 3 significant figures. The ratio of the difference value between two determination results and the arithmetic mean value shall not exceed 10%.

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