

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 952-2018

**Soil and sediment - Determination of polybrominated diphenyl
ethers - Gas chromatography mass spectrometry**

土壤和沉积物多溴二苯醚的测定气相色谱-质谱法

(English Translation)

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Foreword

To implement the Environmental Protection Law of the People's Republic of China, protect the environment, protect human health, and regulate soil and sediment this standard is developed for the determination of polybrominated diphenyl ether.

This standard specifies gas chromatography-mass spectrometry for the determination of polybrominated diphenyl ethers in soils and sediments.

Appendix A of this standard is a normative appendix, and Appendix B and Appendix C are informative appendices.

This standard is the first release.

This standard is formulated by the Environmental Monitoring Department of the Ministry of Ecology and Environment and the Science and Technology Standards Department.

This standard was drafted. National Environmental Analysis and Testing Center, Environmental Protection External Cooperation Center of the Ministry of Ecology and Environment.

This standard is verified by. Hubei Environmental Monitoring Center Station, Guangdong Environmental Monitoring Center, Zhejiang Environmental Monitoring Center, Ningbo Guoke Monitoring Technology Co., Ltd., China National Academy of Environmental Sciences, State Key Laboratory of Environmental Benchmarks and Risk Assessment, and Island Tianjin Enterprise Management (China) Co., Ltd. Beijing Analysis Center.

This standard is approved by the Ministry of Ecology and Environment on July 29, 2018.

This standard has been implemented since December 1, 2018.

This standard is explained by the Ministry of Ecology and Environment.

1 Determination of soil and sediment PBDEs by gas chromatography-mass spectrometry

Warning. The organic solvents and reference materials used in the experiment are toxic and hazardous substances, standard solution preparation and sample preparation.

The process should be carried out in a fume hood; protective equipment should be worn as required to avoid direct contact with skin and clothing.

1 Scope of application this standard specifies gas chromatography-mass spectrometry for the determination of polybrominated diphenyl ethers in soils and sediments.

This standard applies to the determination of eight PBDE congeners in soils and sediments.

When the sample volume is 10.0 g and the volume is 1.0 ml, the method for determining the tris-heptabromodiphenyl ether determined by this standard is detected.

The limit is 0.1~0.2g/kg, the lower limit of determination is 0.4~0.8g/kg; the detection limit of decabromodiphenyl ether is 2.7g/kg, the lower limit is 10.8 g/kg. See Appendix A for details.

2 Normative references

This standard refers to the following documents or their terms. For undated references, the valid version applies to this standard.

GB 17378.3 Marine monitoring specification Part 3. Sample collection, storage and transport
GB 17378.5 Marine monitoring specification Part 5. Sediment analysis
HJ 494 Water Quality Sampling Technical Guidance
HJ 613 Determination of dry matter and moisture in soils - Gravimetric method
HJ/T 166 Technical Specifications for Soil Environmental Monitoring

3 Principle of the method

Extracting PBDE from soil or sediment by pressurized fluid extraction or Soxhlet extraction, the extract is purified, After concentration and constant volume, it was separated by gas chromatography and detected by mass spectrometry. Qualitative according to retention time, fragment ion mass-to-charge ratio and abundance ratio, Quantitative isotope dilution method using selective ion detection.

4 Reagents and materials

Analytically pure reagents that meet national standards are used for analysis, unless otherwise stated.

water.

4.1

Dichloromethane (CH_2Cl_2). pesticide residue grade.

4.2

Hexane (C_6H_{14}). pesticide residue grade.

4.3

Methanol (CH_3OH). pesticide residue grade.

4.4

Acetone (CH_3COCH_3). pesticide residue grade.

4.5

Sodium hydroxide (NaOH). excellent grade.

4.6

Sulfuric acid. $\rho(\text{H}_2\text{SO}_4) = 1.84 \text{ g/ml}$.

24.7 Nitric acid. $\rho(\text{HNO}_3) = 1.42 \text{ g/ml}$.

4.8

Nitric acid solution. 19.

4.9

Sodium hydroxide solution. $\rho(\text{NaOH}) = 50 \text{ g/L}$.

Dissolve 50 g of sodium hydroxide (4.5) in a small amount of water and dilute to 1 L.

4.10

Standard stock solution of polybrominated diphenyl ether. $\rho = 20.0 \text{ mg/L}$, including BDE-28, BDE-47, BDE-99, BDE-100, BDE-153, BDE-154 and BDE-183; wherein the concentration of BDE-209 is 200 mg/L . Using decane as a solvent Standard substance preparation. You can also purchase a commercially available certified standard solution directly. For the storage time, please refer to the relevant instructions of the standard solution certificate.

4.11

Polybromodiphenyl ether solution. $\rho = 2.00 \text{ mg/L}$, of which BDE-209 concentration is 20.0 mg/L .

Pipette $100 \mu\text{l}$ of the standard stock solution of PBDE (4.10) into a 1.5 ml injection vial and add $900 \mu\text{l}$ of n-hexane (4.2).

Mix well. Available now.

4.12

Carbon-labeled PBDE standard solution (extract internal standard). $\rho=2.00$ mg/L, including 13C-BDE-28, 13C-BDE-47, 13C-BDE-99, 13C-BDE-100, 13C-BDE-153, 13C-BDE-154 and 13C-BDE-183; among them, 13C-BDE-209 the concentration was 20.0 mg/L. The solvent is decane, a commercially available certified standard solution.

4.13

Carbon Labeling PCB-209 Standard stock solution. $\rho=50.0$ mg/L, solvent is decane, commercially available certified standard solution.

4.14

Carbon Label PCB-209 Use Solution (Injection Internal Standard). $\rho = 5.00$ mg/L.

Pipette 1.00 ml of carbon-labeled PCB-209 standard stock solution (4.13) into a 10 ml volumetric flask with n-hexane (4.2) Make up to the mark and mix. Store under 4 °C for 3 months.

4.15 4,4'-DDT (p, p'-DDT) standard stock solution. $\rho = 100$ mg/L, the solvent is methanol, commercially available certified standard solution.

4.16 4,4'-DDT standard use solution. $\rho=5.00$ mg/L.

Pipette 50 μ l of p,p'-DDT standard stock solution (4.15) into a 1.5 ml sample vial and add 950 μ l of n-hexane (4.2).

Mix well. Available now.

4.17

Decafluorotriphenylphosphine (DFTPP) standard stock solution. $\rho=1000$ mg/L, solvent is methanol, commercially available certified standard liquid.

4.18

Standard use solution of decafluorotriphenylphosphine. $\rho=50.0$ mg/L.

Pipette 50 μ l of decafluorotriphenylphosphine standard stock solution (4.17) into a 1.5 ml sample vial and add 950 μ l of n-hexane (4.2).

Mix well. Available now.

4.19

anhydrous sodium sulfate (Na_2SO_4). excellent grade pure.

It was fired in a muffle furnace at 450 °C for 4 h, cooled to room temperature, placed in a ground glass bottle, and stored in a desiccator.