

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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**Soil and sediment - Determination of 20 polybrominated
biphenyls - Gas chromatography-high resolution mass
spectrometry**

土壤和沉积物 20种多溴联苯的测定 气相色谱-高分辨质谱法

(English Translation)

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1 Scope of application

This standard specifies the gas chromatography-high resolution mass spectrometry method for the determination of polybrominated biphenyls in soil and sediment.

This standard applies to 2-monobromobiphenyl, 3-monobromobiphenyl, 2,5-dibromobiphenyl, 2,6-dibromobiphenyl, 4,4'-dibromobiphenyl in soil and sediment Benzene, 2,4,6-Tribromobiphenyl, 2,2',4,5'-Tetrabromobiphenyl, 2,2',5,5'-Tetrabromobiphenyl, 3,3',4,4'-tetrabromobiphenyl, 2,2',4,5,5'-pentabromobiphenyl, 2,2',4,5,6-Pentabromobiphenyl, 2,2',4,4',5,5'-hexabromobiphenyl, 2,2',4,4',5,6'-Hexabromobiphenyl, 2,3,3',4,4',5-hexabromobiphenyl, 3,3',4,4',5,5'-Hexabromobiphenyl, 2,2',3,4,4',5,5'-heptabromobiphenyl, 2,2',3,3',4,4',5,5'-octabromobiphenyl Benzene, 2,3,3',4,4',5,5',6-octabromobiphenyl, 2,2',3,3',4,4',5,5',6-Nonabromobiphenyl, 2,2',3,3',4,4',5,5',6,6-Determination of 20 kinds of polybrominated biphenyls such as

'-decabromobiphenyl.

When the sampling amount is 10.0 g and the constant volume is 50 μl , the detection limit of the method for the determination of mono- to deca-bromobiphenyls in this standard is 0.01 $\mu\text{g/kg}$ ~ 0.1 $\mu\text{g/kg}$, the lower limit of determination is 0.04 $\mu\text{g/kg}$ ~0.4 $\mu\text{g/kg}$. See Appendix A for details.

2 Normative references

This standard refers to the following documents or clauses thereof. For dated references, only the dated version applies to this standard.

For undated references, the latest edition (including all amendments) applies to this standard.

GB 17378.3 Marine Monitoring Specification Part 3: Sample Collection, Storage and Transportation GB 17378.5 Marine Monitoring Specification Part 5: Sediment Analysis HJ/T 91 Technical Specification for Surface Water and Sewage Monitoring HJ/T 166 Technical Specification for Soil Environment Monitoring HJ 442.4 Technical Specifications for Environmental Monitoring in Offshore Seas Part 4 Monitoring of Sediments in Offshore Seas HJ 494 Water Quality Sampling Technical Guide HJ 613 Gravimetric Method for Determination of Soil Dry Matter and Moisture

3 Principles of the method

The target compounds such as 2-bromobiphenyl in soil or sediment samples were extracted, concentrated, purified and fixed with organic solvents, and then analyzed by gas chromatography.

Spectroscopic-high-resolution mass spectrometer separation and detection, qualitative determination based on retention time, characteristic ion abundance ratio, and quantification by isotope dilution internal standard method.

4 Reagents and Materials

Unless otherwise specified, analytical reagents that meet national standards are used in the analysis, and the experimental water is freshly prepared without target compounds.

Distilled water or water prepared by pure water equipment.

4.1

Acetone ($\text{C}_3\text{H}_6\text{O}$). pesticide residue grade.

4.2

Toluene (C_7H_8). pesticide residue grade.

4.3

n-hexane (C₆H₁₄). pesticide residue grade.

4.4

Methanol (CH₃OH). pesticide residue grade.

4.5

Dichloromethane (CH₂Cl₂). pesticide residue grade.

4.6

Nonane (C₉H₂₀). pesticide residue grade.

4.7

Hydrochloric acid (HCl). excellent grade, =1.18 g/ml.

4.8

Sulfuric acid (H₂SO₄). excellent grade pure, =1.84 g/ml.

4.9

Sodium hydroxide (NaOH). excellent grade pure.

4.10

Potassium hydroxide (KOH). excellent grade pure.

4.11

Anhydrous sodium sulfate (Na₂SO₄). excellent grade.

Ignited at 450 °C for 4 h, cooled to 150 °C and transferred to a desiccator. After cooling to room temperature, it was put into a glass reagent bottle and kept in a desiccator.

live.

4.12

Acetone-n-hexane mixed solvent.

Acetone (4.1) and n-hexane (4.3) were mixed in a volume ratio of 1.9.

4.13

Hydrochloric acid solution.

Hydrochloric acid (4.7) and water were mixed in a volume ratio of 1.5.

4.14

Sodium hydroxide solution. (NaOH)=40 g/L.

Weigh 4 g of sodium hydroxide (4.9), dissolve it in a small amount of water, and dilute to 100 ml.

4.15

Potassium hydroxide solution. (KOH)=112 g/L.

Weigh 11.2 g of potassium hydroxide (4.10), dissolve it in a small amount of water, and dilute to 100 ml.

4.16

Standard stock solution of polybrominated biphenyls. =50 µg/ml.

For the standard solution of polybrominated biphenyls prepared with nonane (4.6) or other solvents, see Table B.1 in Appendix B. Available for direct purchase Standard solution, refrigerated, sealed, and protected from light below 4 °C, or stored in accordance with the requirements of the standard solution certificate.

4.17

Standard solution for polybrominated biphenyls. =1.0 µg/ml.

Dilute the standard stock solution of polybrominated biphenyls (4.16) with toluene (4.2) or nonane (4.6), refrigerate, seal, and protect from light for 1 a at below 4 °C.

4.18

Extract internal standard stock solution. =50 µg/ml.

Select isotope-labeled compounds as internal standards for extraction, see Table B.1 in Appendix B for details. Commercially available certified standard solutions can be purchased directly, 4 °C the following refrigerated, sealed, protected from light, or stored in accordance with the requirements of the standard solution certificate.

4.19

Internal standard extraction solution. =100 ng/ml.

Dilute the extraction internal standard stock solution (4.18) with toluene (4.2) or nonane (4.6), refrigerate it below 4 °C, seal it, and store it in the dark for 1 a.

4.20

Internal standard stock solution for injection. =2000 µg/ml.

Select isotope-labeled compounds as the injection internal standard, see Table B.1 in Appendix B. Commercially available certified standard solutions can be purchased directly, 4 °C the following refrigerated, sealed, protected from light, or stored in accordance with the requirements of the standard solution certificate.

4.21

Internal standard solution for injection. =100 ng/ml.

Dilute the injection internal standard stock solution (4.20) with toluene (4.2) or nonane (4.6),