

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

HJ 909-2017

**Water quality--Determination of polybrominated diphenyl
ethers--Gas chromatography mass spectrometry**

水质 多溴二苯醚的测定 气相色谱-质谱法

(English Translation)

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Foreword

In order to implement the "Law of the People's Republic of China on Environmental Protection" and the "Law of the People's Republic of China on Prevention and Control of Water Pollution", protect the environment, Protection of human health, regulate the determination of PBDE in water, the development of this standard.

This standard specifies the determination of surface water, groundwater, industrial waste water and domestic sewage by polybrominated diphenyl ether gas chromatography - quality Spectrum method.

This standard Appendix A normative appendix, Appendix B ~ Appendix C is an informative annex.

This standard is released for the first time.

This standard by the Environmental Protection Department of Environmental Monitoring Division and Science and Technology Standards Division to develop.

This standard was drafted. National Environmental Analysis Test Center, Environmental

Protection Department of Environmental Cooperation Center.

This standard verification unit. Hubei Provincial Environmental Monitoring Center Station, Environmental Monitoring Center of Guangdong Province, Zhejiang Province Environmental Monitoring Center, Ningbo Guoke Monitoring Technology Co., Ltd., China State Environmental Sciences Research Institute of Environmental Criteria and Risk Assessment State Key Laboratory and Island Tianjin Enterprise Management (China) Co., Ltd. Beijing Analysis Center.

This standard MEP approved on December 29, 2017.

This standard since April 1, 2018 come into operation.

This standard is interpreted by the MEP.

Water quality - Determination of polybrominated diphenyl ethers - Gas chromatography - mass spectrometry Warning. The internal standards and standard samples used in the experiment are all toxic and hazardous compounds, the solution preparation should be in the fume hood Conduct, operation should be required to wear protective equipment, to avoid contact with skin and clothing.

1 Scope of application

This standard specifies the determination of polybrominated diphenyl ethers in water by gas chromatography - mass spectrometry.

This standard applies to surface water, groundwater, industrial wastewater and domestic sewage eight kinds of PBDE determination of similar substances.

When the sample volume is 1 L, the final volume of 1 ml, the standard determination of three to seven brominated diphenyl ether method was detected the limit of detection ranged from 0.5 ng/L to 1.6 ng/L. The detection limit of decabromodiphenyl ether was 0.02 µg/L. The detection limit of three to seven brominated diphenyl ethers the lower limit of determination is 2.0 ng/L ~ 6.4 ng/L, and the lower limit of determination of decaBDE is 0.08 µg/L. See Appendix A for details.

2 Normative references

This standard references the following documents or the terms. For undated references, the effective version applies to this book standard.

HJ/T 91 Technical Specifications for Surface Water and Sewerage Monitoring HJ/T 164 Groundwater Environmental Monitoring Technical Specifications

3 Method principle

With dichloromethane extraction of water samples of polybrominated diphenyl ether, the extract was dehydrated, concentrated, purified, after volume, with gas color Spectral separation, mass spectrometry. According to the retention time, fragment ion mass ratio and its abundance than qualitative, internal standard method.

4 Reagents and materials

Unless otherwise specified, the analysis of the use of analytical reagents in line with national standards, the experimental water is not pure water.

4.1

Dichloromethane (CH_2Cl_2). pesticide residues.

4.2

n-hexane (C_6H_{14}). pesticide grade.

4.3

methanol (CH_3OH). pesticide grade.

4.4

Sulfuric acid (H_2SO_4). $\rho(\text{H}_2\text{SO}_4) = 1.84 \text{ g/ml}$.

4.5

Hydrochloric acid (HCl). $\rho(\text{HCl}) = 1.19 \text{ g/ml}$.

4.6

Sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$). excellent grade pure.

4.7

PBDE mixed standard solution. $\rho = 20.0 \text{ mg/L}$, including BDE-28, BDE-47, BDE-99, BDE-100, BDE-153, BDE-154 and BDE-183 (BDE-209 at a concentration of 200 mg/L). With n-hexane as solvent, using standard Material preparation, below 4°C sealed dark for six months. You can also buy a certified standard solution, see the standard storage time Liquid certificate related instructions.

4.8

Purification of internal standard solution. $\rho = 2.00 \text{ mg/L}$ including 13C-BDE-28, 13C-BDE-47, 13C- BDE- 99, 13C- BDE- 100, 13C-BDE-153, 13C-BDE-154 and 13C-BDE-183 at a 13C-BDE-209 concentration of 20 mg/L . Commercial permit standards Solution, the solvent is nonane.

4.9

injection of internal standard stock solution. $\rho = 50.0 \text{ mg/L}$, ^{13}C -PCB-209, a commercially available certified standard solution, the solvent is nonane.

4.10

Polybrominated diphenyl ether mixed standard solution I. $\rho = 2.00 \text{ mg/L}$ ($\rho_{\text{BDE-209}} = 20 \text{ mg/L}$).

Pipette 1.00 ml polybrominated diphenyl ether mixed standard solution (4.7) into a 10 ml brown volumetric flask with n-hexane (4.2) Set the volume to mark, mix well. Sealed at 4°C for 3 months.

4.11

Polybrominated diphenyl ether mixed standard solution II. $\rho = .200 \mu\text{g/L}$ ($\rho_{\text{BDE-209}} = .2000 \mu\text{g/L}$).

Pipette 100 μl of PBDE mixed standard solution I (4.10) and 900 μl of n-hexane (4.2) to 1.5 ml Brown sample vial, mix well. Pro use now with.

4.12

injection internal standard solution. $\rho = 5.00 \text{ mg/L}$.

Pipette 1.00 ml sample internal standard stock solution (4.9) to 10 ml brown volumetric flask, n-hexane (4.2) to volume Line, mix well. Sealed at 4°C for 3 months.

4.13

anhydrous sodium sulfate (Na_2SO_4). excellent grade pure.

Burning at 400°C 4 h, cooled into the ground glass jar, placed in a desiccator to save.

4.14

Quartz cotton. Before use, burning at 350°C 2 h, sealed and stored.

4.15

Sodium hydroxide solution. $\rho (\text{NaOH}) = 0.05 \text{ g/ml}$.

Take 50 g of sodium hydroxide (excellent grade pure) dissolved in a small amount of water, diluted to 1 L.

4.16

Silica gel. about $75 \mu\text{m} \sim 180 \mu\text{m}$ (80 mesh $\sim$.200 mesh) chromatography column packed with silica gel.

In a beaker followed by methanol (4.3) and dichloromethane (4.1) wash, volatile completely, spread out in the evaporating dish, Thickness less than 10 mm. 130°C drying 16 h, then placed in a desiccator cooling 30 min, into the conical flask, stored in a desiccator.