

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 715-2014

**Water quality. Determination of polychlorinated biphenyls
(PCBs). Gas chromatography mass spectrometry**

水质 多氯联苯的测定 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 and the Law of the People's Republic of China on Water Pollution Prevention and Control, to protect the environment, to ensure human health, standardize the determination method of polychlorinated biphenyls in water, and formulate this standard.

This standard specifies gas chromatography-mass spectrometry for the determination of 18 polychlorinated biphenyls in water.

This standard is the first release.

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

This standard was formulated by the Science and Technology Standards Department of the Ministry of Environmental Protection.

This standard is mainly drafted by. Harbin Environmental Monitoring Center Station.

The standard verification unit. Heilongjiang Environmental Monitoring Center Station, Dalian Environmental Monitoring Center, Changchun City Environmental Monitoring Xinzhan, Environmental Monitoring Center Station of Anshan City, Environmental Monitoring Center Station of Qiqihar City and Environmental Monitoring Center of Daqing City.

This standard was approved by the Ministry of Environmental Protection on November 27, 2014.

This standard has been implemented since January 1, 2015.

This standard is explained by the Ministry of Environmental Protection.

1 Determination of water quality polychlorinated biphenyls - Gas chromatography - mass spectrometry Warning. PCBs are toxic, carcinogenic, teratogenic, mutagenic, avoiding entrance and contact skin skin. Organic solvents such as n-hexane used in the analysis process are toxic to the human body and should be worn by analysts.

Gloves and respirator with activated carbon layer and operate in a fume hood to minimize and reduce breathing Contact and skin contact.

1 Scope of application

This standard specifies gas chromatography-mass spectrometry for the determination of 18 polychlorinated biphenyls in water.

This standard applies to the determination of 18 polychlorinated biphenyls in surface water, groundwater, industrial wastewater and domestic sewage.

When the sampling amount is 1L, the detection limit of the method is 1.4 to 2.2 ng/L, and the lower limit of determination is 5.6 to 8.8 ng/L. See details Appendix A.

2 Normative references

The contents of this standard refer to the following documents or their terms. For undated references, the valid version is appropriate.

Used in this standard.

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

3 Principle of the method

The polychlorinated biphenyls in the sample are extracted by liquid-liquid extraction or solid phase extraction, and the extract is dehydrated, concentrated, purified and fixed.

The volume was separated and determined by gas chromatography-mass spectrometry. According to retention time, fragment ion mass-to-charge ratio and different ion abundance

ratio Sex, internal standard method quantitative.

4 Interference and elimination

The chromatographic peaks of other PCB congeners coexisting in the sample will interfere with the target compound, and 50% can be selected.

Confirm with an octyl/50% methyl siloxane column or other equivalent column or use a longer capillary column.

5 Reagents and materials

Unless otherwise stated, the analysis uses a superior purification reagent that meets national standards, and the experimental water is a freshly prepared steam.

Distilled water.

5.1

n-hexane (C₆H₁₄). pesticide residue grade.

5.2

Methanol (CH₄O). pesticide residue grade.

5.3

ethyl acetate (C₄H₈O₂). pesticide residue grade.

5.4

Ethyl ether (C₂H₆O). pesticide residue grade.

5.5

Acetone (C₃H₆O). pesticide residue grade.

5.6

n-hexane/ethyl acetate solution. 1 1.

5.7

Sodium chloride (NaCl). heated at 450 ° C for 4 h, placed in a desiccator to cool to room temperature, sealed and stored in a clean test 2 dose bottles.

5.8

anhydrous sodium sulfate (Na₂SO₄). heated at 450 ° C for 4 h, placed in a desiccator to cool to room temperature, sealed and stored in dry the net is in the reagent bottle.

5.9

sodium thiosulfate (Na₂S₂O₃).

5.10

Hydrochloric acid. $\rho(\text{HCl}) = 1.18 \text{ g/ml}$.

5.11

Hydrochloric acid solution. 1 1.

5.12

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

5.13

sodium hydroxide solution. $\rho(\text{NaOH}) = 0.4 \text{ g/ml}$.

Weigh 40g of sodium hydroxide, dilute to 100 ml with water, and mix.

5.14

sodium chloride solution. $\rho(\text{NaCl}) = 0.05 \text{ g/ml}$.

Weigh 5 g of sodium chloride (NaCl), dilute to 100 ml with water, and mix.

5.15

Eluent 1. 6 94 diethyl ether/n-hexane mixed solution.

5.16

Eluent 2. 1 9 acetone/n-hexane mixed solution.

5.17

standard stock solution. $\rho = 1.0 \text{ } \mu\text{g/ml}$, the solvent is n-hexane.

Commercially available certified standard solutions can be purchased directly. Including PCB28, PCB52, PCB101, PCB81, PCB77, PCB123, PCB118, PCB114, PCB138, PCB105, PCB153, PCB126, PCB167, PCB156, PCB157, PCB180, PCB169, PCB189. Sealed below 4°C and protected from light, or refer to the manufacturer's recommended storage conditions.

5.18

Internal standard stock solution (IS). PCB77-d6, PCB156-2 , 6,6 -d3, $\rho=100.0 \text{ } \mu\text{g/ml}$.

The certified standard solution can be purchased directly or prepared with standard materials and diluted with n-hexane. Below 4 ° C, sealed, avoid Save light, or refer to the manufacturer's recommended storage conditions. Other isotopically labeled internal standards can also be used.

5.19

internal standard use solution. $\rho = 20.0 \text{ } \mu\text{g/ml}$.

The internal standard stock solution (5.18) was diluted with n-hexane.