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HJ

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

HJ 591-2010

Replacing GB/T 8972-1988

**Water quality. Determination of pentachlorophenol by gas
chromatography**

水质 五氯酚的测定 气相色谱法

(English Translation)

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Table of Contents

Foreword	
1 Scope of application	
2 Principle of the method	
3 Reagents and materials	
4 Instruments and equipment	
4.2 Column	
5 Samples	
5.1 Sample collection and preservation	
5.2 Preparation of samples	
6 Analysis steps	
6.1 Chromatographic conditions	
6.2 Drawing of the working curve	
6.3 Chromatogram of standard solution	
6.4 Determination	
6.5 Blank test	
7 Calculation and representation of results	
7.1 Calculation of results	
7.2 Results representation	
8 Precision and accuracy	
8.1 Precision	
8.2 Accuracy	
9 Quality Assurance and Quality Control	
9.1 Qualitative analysis	
9.2 Blank test	
9.4 Intermediate mass concentration test	
9.5 Matrix addition	

Foreword

To protect the environment and protect the human body in order 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 this standard is established for the determination of health and standardization of pentachlorophenol in

water.

This standard specifies the gas chromatographic method for the determination of pentachlorophenol and pentachlorophenolate in water.

This standard is a revision of the "Gas Chromatography for the Determination of Pentachlorophenol in Water" (GB 8972-88).

This standard was first published in 1988. The original standard was drafted by the Ministry of Railways Labor Health Research Institute. This is the first revision. Revision the main contents are as follows.

- Expanded the scope of application;
- Added capillary column analysis method;
- modified the quantitative method;
- Added provisions for quality control and quality assurance.

From the date of implementation of this standard, the national environmental protection standard "Water quality approved and issued by the former National Environmental Protection Agency on March 26, 1988.

Determination of pentachlorophenol gas chromatography (GB 8972-88) is abolished.

Appendix A of this standard is an informative annex.

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

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

This standard is verified by. Jinan Environmental Protection Monitoring Station, Zibo Environmental Monitoring Station, Weifang Environmental Monitoring Center Station, Qingdao Institute of Technology College of Environmental and Municipal Engineering, Agricultural Products Supervision and Inspection Center of the Ministry of Agriculture (Qingdao).

This standard was approved by the Ministry of Environmental Protection on October 21, 2010.

This standard has been implemented since January 1, 2011.

This standard is explained by the Ministry of Environmental Protection.

Water quality - Determination of pentachlorophenol - Gas chromatography

1 Scope of application

This standard specifies the gas chromatographic method for the determination of pentachlorophenol and pentachlorophenolate in water.

This standard applies to the determination of pentachlorophenol and pentachlorophenolate in surface water, groundwater, seawater, domestic sewage and industrial wastewater.

When the sample volume is 100 ml, the detection limit of capillary column gas chromatography is 0.01 µg/L, and the lower limit of determination is 0.04 µg/L.

The limit is 5.00 µg/L; the detection limit of packed column gas chromatography is 0.02 µg/L, and the lower limit of determination is 0.08 µg/L.

2 Principle of the method

The pentachlorophenolate in the sample is converted to pentachlorophenol under acidic conditions, extracted with n-hexane, and then back extracted with potassium carbonate solution to give the pentachlorophenol in the organic phase is converted to pentachlorophenolate into an alkaline aqueous solution. Adding acetic anhydride and pentachlorophenolate to an alkaline aqueous solution for derivatization should produce pentachlorophenyl acetate. After extraction with n-hexane, the measurement was carried out using a gas chromatograph with an electron capture detector.

3 Reagents and materials

Analytical purification reagents conforming to national standards were used for the analysis, and the experimental water was freshly prepared distilled water unless otherwise stated.

3.1

n-hexane (C₆H₁₄). pesticide residue level.

3.2

Acetic anhydride [(CH₃CO)₂O].

3.3

Methanol (CH₃OH). pesticide residue grade.

3.4

Copper sulfate (CuSO₄).

3.5

sodium thiosulfate (Na₂S₂O₃ · 5H₂O).

3.6

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

3.7

Sulfuric acid solution. 19.

3.8

Potassium carbonate solution (K_2CO_3). $c = 0.1 \text{ mol/L}$.

Weigh 13.8 g of anhydrous potassium carbonate and dissolve it in 1 000 ml of water.

3.9

anhydrous sodium sulfate (Na_2SO_4).

After burning at 400°C for 2 h in a muffle furnace, after cooling, it was stored in a ground glass bottle and sealed.

3.10

Pentachlorophenol standard stock solution. $\rho = 1.00 \text{ mg/ml}$.

Store at 4°C in cold storage. Commercially available certified standards can be used.

3.11

Pentachlorophenol standard intermediate solution. $\rho = 100.0 \text{ }\mu\text{g/ml}$.

Accurately transfer $100.0 \text{ }\mu\text{l}$ of pentachlorophenol standard stock solution (3.10) to 1 ml with methanol.

3.12

Pentachlorophenol standard use solution. $\rho = 1.00 \text{ }\mu\text{g/ml}$.

Accurately remove $10.00 \text{ }\mu\text{l}$ of pentachlorophenol standard intermediate (3.11) and dilute to 1 ml with methanol.

3.13

Carrier gas. High purity nitrogen [purity $\geq 99.99\%$ (volume fraction)].

4 Instruments and equipment

4.1

Gas Chromatograph. with an electron capture detector.

4.2 Column

Packed column. made of hard glass or stainless steel, 1.5 to 2.5 m long, 3 to 4 mm in inner diameter, 1.5% phenyl-containing poly-coated Chromsorb W (80-100 mesh) of methyl