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HJ

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

HJ 1049-2019

**Water quality--Determination of 4 nitrophenol compounds
--Liquid chromatography-triple quadrupole mass spectrometry**

水质 4 种硝基酚类化合物的测定 液相色谱-三重四极杆质谱法

(English Translation)

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Table of Contents

Foreword	
1 Scope	
2 Normative references	
3 Methodology	
4 Interference and cancellation	
5 Reagents and materials	
6 Instruments and equipment	
7 Sample	
8 Analysis steps	
9 Results calculation and representation	
10 Precision and accuracy	
11 Quality Assurance and Quality Control	
12 Waste disposal	
Appendix A (Normative) Method detection limit and determination limit	
Appendix B (informative) Mass spectrometer reference conditions	
Appendix C (informative) Precision and accuracy of the method	

Foreword

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 Control", protect the ecology Environment, protect human health, standardize the determination method of nitrophenol compounds in water, and formulate this standard.

This standard specifies the liquid phase determination of 4 nitrophenol compounds in surface water, groundwater, domestic sewage and industrial wastewater.

Chromatography-Triple Quadrupole Mass Spectrometry.

Appendix A to this standard is a normative appendix, and appendixes B to C are informative appendixes.

This standard is issued for the first time.

This standard is formulated by the Department of Eco-Environmental Monitoring, Laws and Standards Department of the Ministry of Ecology and Environment.

This standard was drafted. Sichuan Ecological Environment Monitoring General Station.

Verification units of this standard. Chongqing Ecological Environment Monitoring Center, Guangyuan Environmental Monitoring Center Station, Panzhihua Environmental Monitoring Center Monitoring Center Station, Shengzhou Environmental Monitoring Center Station, Yibin Environmental Monitoring Center Station and Nanchong Environmental Monitoring Center Station.

This standard was approved by the Ministry of Ecology and Environment on October 24, 2019.

This standard will be implemented as of April 24, 2020.

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

Determination of 4 nitrophenol compounds in water quality Liquid chromatography-triple quadrupole mass spectrometry Warning. The standard materials and organic solvents used in the experiment are toxic and hazardous substances, reagent preparation and sample preparation process it should be carried out in a fume hood; wear protective equipment as required to avoid contact with skin and clothing.

1 Scope

This standard specifies liquid chromatography-triple quadrupole mass spectrometry for the determination of four nitrophenol compounds in water.

This standard applies to 2,6-dinitrophenol, 2,4-dinitrophenol, Determination of 4 nitrophenols such as 4-nitrophenol and 2,4,6-trinitrophenol.

When the injection volume is 10 μL , the method detection limits of the four nitrophenol compounds are 0.4 $\mu\text{g/L}$ ~ 0.6 $\mu\text{g/L}$.

The lower limit is 1.6 $\mu\text{g/L}$ to 2.4 $\mu\text{g/L}$. See Appendix A for details.

2 Normative references

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

HJ/T 91 Technical specifications for surface water and sewage monitoring HJ/T 164
Technical Specifications for Groundwater Environmental Monitoring

3 Method principle

The samples were directly injected after filtration or purification, and were separated and detected by liquid chromatography-triple quadrupole mass spectrometry.

Qualitative analysis based on retention time and characteristic ions, quantification by internal standard method.

4 Interference and cancellation

4.1

When matrix interferences exist in the sample, you can optimize the chromatographic conditions, dilute the sample, reduce the injection volume, and Products are reduced or eliminated by pretreatment.

4.2

When there is interference of isomers in the sample, you can improve the resolution by changing the chromatographic conditions or choose a different the secondary mass ion eliminates interference.

4.3

When the interference of coexisting organics in the sample is determined, a part of the interference can be removed by extraction with a mixed solution of n-hexane-dichloromethane.

Take the water phase for analysis.

5 Reagents and materials

Unless otherwise stated, all analytical reagents that meet national standards are used for analysis.

Standard compound of pure water.

5.1

Methanol (CH_3OH). pure by liquid chromatography.

5.2

Dichloromethane (CH_2Cl_2). pure liquid chromatography.

5.3

n-hexane (C_6H_{14}). pure by liquid chromatography.

5.4

Formic acid (HCOOH). pure liquid chromatography.

5.5

Ammonium formate (HCOONH_4). pure liquid chromatography.

5.6

Ammonia. $\rho(\text{NH}_3 \cdot \text{H}_2\text{O}) = 0.91 \text{ g/ml}$, excellent grade pure.

5.7

Ammonium formate-formic acid buffer solution. $c(\text{HCOONH}_4) = 0.01 \text{ mol/L}$, $\text{pH} = 4$.

Accurately weigh 0.315 g of ammonium formate (5.5) and dissolve it in an appropriate amount of water. After dissolving, transfer it to a 500 ml volumetric flask and dilute it with water.

Reduce the volume to the mark, mix well, and add about 70 μL of formic acid (5.4) to adjust the pH to 4.

5.8

n-hexane-dichloromethane mixed solution. 1 2.

Mix with n-hexane (5.3) and dichloromethane (5.2) in a volume ratio of 1. 2.

5.9

Standard stock solution of nitrophenols. $\rho = 100 \text{ mg/L}$.

It can be prepared with standard materials, the purity of standard materials is greater than 99.0%, dissolved in methanol (5.1), frozen below -10°C , Keep away from light. You can also purchase a certified standard solution directly and store it in accordance with the manufacturer's product instructions.

5.10

Standard solution for mixing nitrophenol compounds. $\rho = 5.00 \text{ mg/L}$ (reference concentration).

Transfer an appropriate amount of standard stock solution of nitrophenol compounds (5.9), dilute with methanol (5.1), freeze at -10°C , Keep away from light and can be stored for 3 months.

5.11

Internal standard stock solution. $\rho = 5000 \text{ mg/L}$.

The internal standard is 2,4-dinitrophenol-d3, which can be prepared with standard materials. The purity of the standard material is greater than 98.0%. Methanol (5.1) is used.

Dissolve, store at -10°C or less, and protect from light. Certified standard solutions can also be purchased directly.

5.12

Internal standard use solution. $\rho = .200 \mu\text{g/L}$ (reference concentration).

Dilute the internal standard stock solution (5.11) with methanol (5.1) as needed, freeze it below -10°C , and store it in the dark to protect it.

Deposit for 3 months.