

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

HJ 1150-2020

**Water quality Determination of nitrophenol compounds Gas
chromatography-mass spectrometry**

水质 硝基酚类化合物的测定 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 Water Pollution Prevention Law of the People's Republic of China, and protect the ecology the environment, to protect human health, regulate the determination method of nitrophenol compounds in water, and formulate this standard.

This standard specifies the determination of 12 nitrophenol compounds in surface water, groundwater, domestic sewage, industrial wastewater and seawater Gas chromatography-mass spectrometry.

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

This standard is issued for the first time.

This standard was formulated by the Department of Ecological Environment Monitoring and the Department of Regulations and Standards of the Ministry of Ecology and Environment.

Drafting organization of this standard. Jiangsu Environmental Monitoring Center.

Verifiers of this standard. Zhenjiang Environmental Monitoring Center of Jiangsu Province, Changzhou Environmental Monitoring Center of Jiangsu Province, Taizhou Environmental Monitoring Center of Jiangsu Province Environmental Monitoring Center, Nantong Environmental Monitoring Center of Jiangsu Province, and Huaian Environmental Monitoring Center of Jiangsu Province.

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

1 Water quality Determination of nitrophenolic compounds Gas chromatography-mass spectrometry

Warning. The standard substances and organic solvents used in the experiment are toxic and hazardous substances. The reagent preparation and sample pretreatment process it should be carried out in a fume hood; protective equipment should be worn as required during operation to avoid contact with skin and clothing.

1 Scope of application this standard specifies the gas chromatography-mass spectrometry method for the determination of nitrophenol compounds in water.

This standard applies to 2-nitrophenol and 3-methyl-2-nitro groups in surface water, groundwater, domestic sewage, industrial wastewater and seawater.

Phenol, 4-methyl-2-nitrophenol, 5-methyl-2-nitrophenol, 2,5-dinitrophenol, 3-nitrophenol, 2,4-dinitrophenol, 4-Nitrophenol, 12 kinds of 2,6-dinitrophenol, 3-methyl-4-nitrophenol, 6-methyl-2,4-dinitrophenol and 2,6-dimethyl-4-nitrophenol Nitrophenol Determination of compounds.

When the sampling volume is 1000 ml and the sample volume is 1.0 ml, the detection limit of the method for 12 nitrophenol compounds it is 0.2 µg/L~2 µg/L, and the lower limit of determination is 0.8 µg/L~8 µg/L. See Appendix A for details.

2 Normative references

This standard quotes the following documents or their clauses. For undated reference documents, their valid versions are applicable to this standard.

GB 17378.3 Marine Monitoring Specification Part 3: Sample Collection, Storage and Transportation GB/T 14581 Technical Guidelines for Water Quality Sampling in Lakes and Reservoirs HJ/T 91 Technical specification for surface water and sewage monitoring HJ 91.1 Technical Specification for Wastewater Monitoring HJ/T 164 Technical Specification for Groundwater Environmental Monitoring

3 Principles of the method

After the sample is purified by acid-base distribution, under acidic conditions (pH value of 1~2), use liquid-liquid extraction or solid phase extraction the nitrophenol compound is extracted by extraction method, and the extract is dehydrated, concentrated, and fixed to volume, and then separated by gas chromatography and detected by mass spectrometry. according to Retention time, fragment ion mass-to-charge ratio and abundance ratio are qualitative, and internal standard method is used for quantification.

4 Reagents and materials

Unless otherwise specified, analytical reagents that meet national standards are used for analysis, and the experimental water does not contain target compounds. Distilled water or water prepared by pure water equipment.

4.1

Dichloromethane (CH_2Cl_2). pesticide residue level.

4.2

Acetone (CH_3COCH_3). pesticide residue level.

4.3

Methanol (CH_3OH). pesticide residue level.

4.4

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

24.5 Sodium hydroxide (NaOH).

4.6

Anhydrous sodium sulfate (Na_2SO_4).

Bake in a muffle furnace at 400°C for 4 hours, cool to room temperature in a desiccator, and put it in a reagent bottle for sealed storage.

4.7

Sodium chloride (NaCl).

Bake in a muffle furnace at 400°C for 4 hours, cool to room temperature in a desiccator, and put it in a reagent bottle for sealed storage.

4.8

Hydrochloric acid solution. 1 1.

4.9

Hydrochloric acid solution. $c(\text{HCl})=0.02 \text{ mol/L}$.

Measure 1.8 ml of hydrochloric acid (4.4), slowly add it to water, transfer to a 1000 ml volumetric flask, dilute to the mark.

Available for immediate use.

4.10

Sodium hydroxide solution. $c(\text{NaOH})=5.0 \text{ mol/L}$.

Weigh 20.0 g of sodium hydroxide (4.5), dissolve it with water, transfer to a 100 ml

volumetric flask, dilute to the mark. Near Use now.

4.11

Standard material. purity $\geq 99\%$.

2-nitrophenol, 3-methyl-2-nitrophenol, 4-methyl-2-nitrophenol, 5-methyl-2-nitrophenol, 2,5-dinitrophenol, 3- Nitrate Phenol, 2,4-dinitrophenol, 4-nitrophenol, 2,6-dinitrophenol, 3-methyl-4-nitrophenol, 6-methyl-2,4-dinitrophenol Phenol and 2,6-Dimethyl-4-nitrophenol.

4.12

Standard stock solution. $\rho = 1000 \text{ mg/L}$.

Weigh 50 mg (accurate to 0.1 mg) of nitrophenol compound standard materials (4.11), and use a small amount of methanol (4.3) Dissolve, transfer to a 50 ml brown volumetric flask, dilute with dichloromethane (4.1) to the mark, and mix. The standard solution Frozen and protected from light below -10°C , it can be stored for half a year. It is also possible to directly purchase commercially available certified standard solutions and keep them in accordance with the instructions.

4.13

Standard liquid. $\rho = 200 \text{ mg/L}$.

Dilute the standard stock solution (4.12) with dichloromethane (4.1). It can be stored for 2 months at 4°C in the dark and airtight.

4.14

Internal standard stock solution. $\rho = 2000 \text{ mg/L}$.

Naphthalene-d8 and acenaphthylene-d10 should be used as internal standards for nitrophenol compounds. Commercially available certified standard solutions should be kept as per the instructions.

4.15

Internal standard liquid. $\rho = 500 \text{ mg/L}$.

Dilute the internal standard stock solution (4.14) with dichloromethane (4.1).

4.16

Decafluorotriphenylphosphine (DFTPP) solution. $\rho = 1000 \text{ mg/L}$. Commercially available certified standard solution, guaranteed according to the instruction time Save.

4.17

Decafluorotriphenylphosphine use solution. $\rho = 50 \text{ mg/L}$.

Dilute the decafluorotriphenylphosphine (DFTPP) solution (4.16) with dichloromethane (4.1).