

ICS 13.060
CCS Z 16

HJ

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

HJ 676-2013

**Water quality. Determination of phenolic compounds. Liquid
liquid extraction gas chromatography**

水质 酚类化合物的测定 液液萃取 气相色谱法

(English Translation)

Issued on: January 1, 1970

Implemented on: January 1, 1970

Issued by: Ministry of Ecology and Environment

Table of Contents

Foreword

1 Scope of application

2 Normative references

3 Principle of the method

5 Reagents and materials

5.1 Experimental water

5.12 Sodium chloride

5.13 Anhydrous sodium sulfate

5.14 Standard solution of phenolic compound. $\rho=500\sim 2500$ mg/L

6 Instruments and equipment

7 Samples

7.1 Sample Collection and Storage

12 Shake the separatory funnel for about 3 to 5 minutes, and pay attention to deflation.
Rest

13 Pentachlorophenol 2.0 5.0 10.0 25.0 50.0 100

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, this standard is formulated to ensure human health and to regulate the determination of phenolic compounds in water.

This standard specifies liquid-liquid extraction/gas for the determination of phenolic compounds in surface water, groundwater, domestic sewage and industrial wastewater.

Phase chromatography.

This standard is the first release.

Appendix A, Appendix B and Appendix C of this standard are informative annexes.

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

This standard is mainly drafted by. Ningbo Environmental Monitoring Center.

This standard is verified by. Zhoushan Marine Ecological Environment Monitoring Station, Hangzhou Environmental Monitoring Center Station, Taizhou Environment Monitoring

Center Station, Jiaxing Environmental Protection Monitoring Station, Huzhou Environmental Protection Monitoring Center Station and Jinhua Environmental Monitoring Center Station.

This standard was approved by the Ministry of Environmental Protection on November 21, 2013.

This standard has been implemented since February 1, 2014.

This standard is explained by the Ministry of Environmental Protection.

Water quality - Determination of phenolic compounds - Liquid-liquid extraction/gas chromatography Warning. The solvents and reagents used in the test are all toxic. The sample preparation process should be carried out in a fume hood.

In operation, wear protective equipment as required to avoid direct contact between skin and clothing.

1 Scope of application

This standard specifies liquid-liquid extraction/gas chromatography for the determination of phenolic compounds in water.

This standard applies to surface water, groundwater, domestic sewage and industrial wastewater in phenol, 3-cresol, 2,4-xylene, 2-Chlorophenol, 4-chlorophenol, 4-chloro-3-cresol, 2,4-dichlorophenol, 2,4,6-trichlorophenol, pentachlorophenol, 2-nitrophenol, 4-nitrophenol 2,4-Determination of 13 phenolic compounds such as dinitrophenol and 2-methyl-4,6-dinitrophenol.

When the sampling volume is 500mL, the method detection limits and lower limit of determination of 13 phenolic compounds are shown in Table 1.

Table 1 Method detection limit and lower limit of measurement unit. µg/L

Compound name	detection limit	lower limit	Compound name	detection limit	lower limit
Phenol	0.5	2.0	2,4,6-trichlorophenol	1.2	4.8
3-cresol	0.5	2.0	pentachlorophenol	1.1	4.4
2,4-xylene	0.7	2.8	2-nitrophenol	1.1	4.4
2-Chlorophenol	1.1	4.4	4-Nitrophenol	1.2	4.8
4-chlorophenol	1.4	5.6	2,4-dinitrophenol	3.4	13.6
4-chloro-3-cresol	0.7	2.8			
2-methyl-4,6-dinitrophenol	3.1	12.4	2,4-dichlorophenol	1.1	4.4

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.

GB/T 14581 Water quality lake and reservoir sampling technical guidance HJ/T 91 Surface Water and Wastewater Monitoring Technical Specifications HJ/T 164 Technical

Specifications for Groundwater Environmental Monitoring HJ/T 493 Technical Regulations for Preservation and Management of Water Quality Samples

3 Principle of the method

Under acidic conditions (pH4 interference and elimination

4.1 There may be other organic matter interference measurement in the water sample, which can be purified by alkaline aqueous solution, or by changing color.

Spectral conditions, double column characterization or mass spectrometry were further confirmed.

4.2 There may be a memory effect after measuring a high concentration sample, which can be analyzed by blank sample until the target in the blank sample the next sample can only be analyzed if the concentration of the compound is below the lower limit of the assay.

5 Reagents and materials

Analytical purification reagents that meet national standards were used for analysis unless otherwise stated.

5.1 Experimental water

Secondary distilled water, commercially available purified water or organic-free water prepared by pure water equipment. Should be tested by blank test before use to confirm that there is no interference peak in the retention time interval of the target compound or that the target compound concentration is lower than Method detection limit.

5.2 sodium hydroxide (NaOH).

5.3 Concentrated hydrochloric acid (HCl). $\rho(\text{HCl}) = 1.19 \text{ g/mL}$.

5.4 Aqueous sodium hydroxide solution. $\rho(\text{NaOH}) = 0.2 \text{ g/mL}$.

Weigh 20g of sodium hydroxide (5.2), dissolve in a small amount of water (5.1), and dilute to 100 mL.

5.5 Hydrochloric acid solution. 1:3 (V/V).

Measure 125 mL of concentrated hydrochloric acid (5.3) and dilute to 500 mL with water (5.1).

5.6 Dichloromethane (CH_2Cl_2). pesticide residue grade.

5.7 Ethyl acetate ($\text{CH}_3\text{COOC}_2\text{H}_5$). pesticide residue grade.

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

5.9 Methanol (CH_3OH). pesticide residue grade.

5.10 Mixed solvent of dichloromethane/ethyl acetate. 1 1 (V/V).

Mix with dichloromethane (5.6) and ethyl acetate (5.7) in a volume ratio of 1.1.

5.11 Mixed solvent of dichloromethane/n-hexane. 2 1 (V/V).

Mix with dichloromethane (5.6) and n-hexane (5.8) in a volume ratio of 2.1.

5.12 Sodium chloride

Bake in a muffle furnace at 400 ° C for 4 h, cool to room temperature and store in a desiccator.

5.13 Anhydrous sodium sulfate

Bake in a muffle furnace at 400 ° C for 4 h, cool to room temperature and store in a desiccator.

5.14 Standard solution of phenolic compound. rho=500~2500 mg/L

A methanol solution containing 13 target phenolic compounds, which can be purchased directly from a certified standard solution or a pure standard material.

Ready. The standard solution can be stored for half a year at 4 °C.

5.15 Carrier gas. nitrogen, purity $\geq 99.999\%$.

5.16 Combustion gas. hydrogen, purity $\geq 99.99\%$.

5.17 Gas. Air, which must remove moisture and organic matter.

6 Instruments and equipment

6.1

Gas Chromatograph. With split/splitless inlet, programmable, with a hydrogen flame detector (FID).

6.2

Analytical column. quartz capillary column, 30m x 0.32mm, film thickness 0.25 μ m, fixed solution 5% phenyl-95% Methyl polysiloxane, or other equivalent column.

6.3

Concentrator. A concentrating device of comparable performance, such as a rotary evaporator, a nitrogen blower, or an organic sample concentrator.

6.4

Balance. Accuracy is 0.1 g.