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HJ

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

HJ 744-2015

**Water quality. Determination of phenols compounds. Gas
chromatography mass spectrometry**

水质 酚类化合物的测定气相色谱-质谱法

(English Translation)

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24.10 Anhydrous sodium sulfate (Na_2SO_4)

51 Strength of 19-60% of 19 pieces of.199.199% of the strength of 198 fragments of 5-9%

68 Strength less than 69 pieces of debris 2% 275 strength of 198 fragments of 10-30%

70 Strength Less than 69 pieces of 2% 365 Strength greater than 198% of debris 1%

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 the Prevention and Control of Water Pollution, Human health, regulate the determination of phenolic compounds in water, the development of this standard.

This standard specifies the determination of phenolic compounds in water by gas chromatography-mass spectrometry.

This standard is the first release.

Appendix A of this standard is a normative appendix, Appendix B is an informative appendix.

This standard is organized by the Ministry of Environmental Protection Science and Technology Standards Division.

The main drafting unit of this standard. Jiangsu Province Environmental Monitoring Center.

The standard verification unit. Taizhou City Environmental Monitoring Center Station, Nanjing Environmental Monitoring Center Station, Chongqing Environmental Monitoring Center Station, Dalian Environmental Monitoring Center, the State Environmental Protection Department of Nanjing Environmental Science Research Institute and Henan Province Environmental Monitoring Center.

The environmental protection department of this standard approved on May 4, 2015.

This standard has been implemented since July 1, 2015.

This standard is explained by the Ministry of Environmental Protection.

Determination of water quality phenolic compounds - Gas chromatography - mass spectrometry WARNING. The solvents and reagents used in the test are subject to toxicity and the sample pretreatment should be carried out in a fume hood, Operation should be required to wear protective equipment, to avoid direct contact with solvents and reagents skin and clothing.

1 Scope of application

This standard specifies the determination of phenolic compounds in water by gas chromatography-mass spectrometry.

This standard applies to surface water, groundwater, domestic sewage and industrial wastewater phenol, 2-chlorophenol, 4-chlorophenol, pentachlorophenol, 2,4-dichlorophenol, 2,6-dichlorophenol, 2,4,6-trichlorophenol, 2,4,5-trichlorophenol, 2,3,4,6-tetrachlorophenol, 4- Nitrophenol, 2-methyl Phenol, 3-cresol, 4-cresol, 2,4-xilenol and other 14 kinds of phenolic compounds. Other phenolic compounds are verified by methods Using this method.

When the sampling volume was 250 ml, the method of detection of 14 phenolic compounds was 0.1 g/L when the selective ion scanning mode was used ~ 0.2 g/L, the lower limit of determination is 0.4 g/L ~ 0.8 g/L, see Appendix A.

2 Normative reference documents

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

Technical guidance for sampling of water quality lakes and reservoirs GB/T 14581 Technical specification for surface water and wastewater monitoring Technical specification for groundwater environmental monitoring Technical specification for preservation and management of water quality samples

3 Principle of the method

Under the acidic condition ($\text{pH} \leq 1$), the phenolic compounds in the water samples were extracted by liquid-liquid extraction or solid-phase extraction.

After derivatization, the samples were separated by gas chromatography-mass spectrometry (GC-MS). The chromatographic retention time and mass spectrometry were characterized by ion characterization.

Or internal standard method quantitative.

4 Reagents and materials

Unless otherwise stated, analytical reagents and experimental water in accordance with national standards are used for analysis.

4.1

ethyl acetate ($\text{CH}_3\text{COOC}_2\text{H}_5$). pure chromatography.

4.2

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

4.3

Acetone (CH_3COCH_3). Chromatographic pure.

4.4

Methanol (CH_3OH). Chromatographic pure.

4.5

dichloromethane (CH_2Cl_2). chromatographic purity.

4.6

Sulfuric acid (H_2SO_4). $\rho(\text{H}_2\text{SO}_4) = 1.84 \text{ g/ml}$.

4.7

Hydrochloric acid (HCl). $\rho(\text{HCl}) = 1.19 \text{ g/ml}$.

4.8

Potassium carbonate (K_2CO_3).

4.9

Sodium hydroxide (NaOH).

24.10 Anhydrous sodium sulfate (Na_2SO_4)

Baked in a muffle oven at 400°C for 4 h, cooled to room temperature, placed in a glass jar, and stored in a desiccator.

4.11 Sodium chloride (NaCl) Baked in a muffle oven at 400°C for 4 h, cooled to room temperature, placed in a glass jar, and stored in a desiccator.

4.12 Pentafluorobenzyl bromide ($\text{C}_7\text{H}_2\text{BrF}_5$).

4.13 Dichloromethane-n-hexane mixed solution. 2 1.

Mixed with dichloromethane (4.5) and n-hexane (4.2) at a volume ratio of 2. 1.

4.14 Dichloromethane-ethyl acetate mixed solution. 4 1.

Mixed with dichloromethane (4.5) and ethyl acetate (4.1) in a 4:1 by volume ratio.

4.15 dichloromethane - ethyl acetate mixed solution. 1:1.

And mixed with methylene chloride (4.5) and ethyl acetate (4.1) in a 1:1 by volume ratio.

4.16 sulfuric acid solution. 1:1.

Measure 50 ml of concentrated sulfuric acid (4.6) and slowly add to 50 ml of water.

4.17 hydrochloric acid solution. $C(HCl) = 0.05 \text{ mol/L}$.

Measure 0.44 ml of concentrated hydrochloric acid (4.7) and slowly add to 100 ml of water.

4.18 Potassium carbonate solution (K_2CO_3). $\rho(K_2CO_3) = 0.1 \text{ g/ml}$.

Weigh 1.0 g of potassium carbonate (4.8) in water and set to 10.0 ml.

4.19 Sodium hydroxide solution. $\rho(NaOH) = 0.4 \text{ g/ml}$ Weigh 40 g of sodium hydroxide (4.9) in water and set to 100 ml.

4.20 Pentafluorobenzyl bromide derivatization reagent Weigh 0.500 g of pentafluorobenzyl bromide (4.12), dissolved in 9.5 ml of acetone (4.3) and stored at 4°C for 2 weeks.

4.21 Phenol compound standard stock solution. $\rho = 100 \text{ mg/L}$ to 2000 mg/L Contains 14 kinds of target phenolic compounds. It can be prepared by diluting pure standard with isopropanol. The standard solution is cold and sealed at 4°C Possession, can be saved for six months. Can also be directly purchased certified standard solution, storage time, see the standard solution certificate of the relevant instructions.

4.22 Phenolic compounds Standard use. $\rho = 10.0 \text{ mg/L}$ the standard stock solution (4.21) was diluted with acetone (4.3).

4.23 Standard stock solution. $\rho = 1000 \text{ mg/L}$ the use of 2,5-dibromotoluene as the determination of methyl phenol, dimethyl phenol, monochlorophenol and dichlorophenol and other relatively low boiling point of phenols the internal standard of the compound, 2,2',5,5'-tetrabromobiphenyl is used as a phenolic compound for the determination of higher boiling point such as trichlorophenol, tetrachlorophenol and nitrophenol the internal standard. Can be directly purchased certified standard solution, or diluted with pure isopropyl alcohol standard material preparation. 4°C under refrigeration, can be stored for six months.

4.24 Standard use of liquid. $\rho = 100 \text{ mg/L}$.

The standard stock solution (4.23) was diluted with n-hexane (4.2).

4.25 Substitute Standard stock solution. $\rho = 2000 \text{ mg/L}$ 2-fluorophenol, 2,4,6-tribromophenol can be used as a substitute. Can be directly purchased certified standard solution, or diluted with isopropyl alcohol pure standard Quasi-material preparation. 4°C under the cold storage, can be stored for six months.