

ICS 13.060
CCS Z 16

HJ

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

HJ 1073-2019

**Water quality--Determination of naphthol--High performance
liquid chromatography**

水质 萘酚的测定 高效液相色谱法

(English Translation)

Issued on: January 1, 1970

Implemented on: June 30, 2020

Issued by: Ministry of Ecology and Environment

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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 naphthol in water, and formulate this standard.

This standard specifies the determination of 1-naphthol (alpha-naphthol) in surface water, groundwater, domestic sewage, industrial wastewater and seawater.

High-performance liquid chromatography of 2-naphthol (beta-naphthol).

Appendix A and Appendix B of this standard are informative appendices.

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. Liaoning North Environmental Detection Technology Co., Ltd.

Verification units of this standard. Liaoning Province Ecological Environment Monitoring

Center, Shandong Province Qingdao Ecological Environment Monitoring Center, Liaoning Province, Shenyang Yang Ecological Environment Monitoring Center, Anshan Environmental Monitoring Center, Liaoning Province, Liaoyang Environmental Monitoring Center, Liaoning Province, and Shenyang Zeer Testing Services Ltd.

This standard was approved by the Ministry of Ecology and Environment on December 31, 2019.

This standard will be implemented from June 30, 2020.

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

Water quality-Determination of naphthol-High performance liquid chromatography Warning. The standard materials and organic solvents used in the experiment are all toxic. The reagent preparation and sample preparation have been processed.

The process should be carried out in a fume hood, and protective equipment should be worn as required to avoid direct contact with skin and clothing.

1 Scope

This standard specifies the HPLC method for the determination of naphthol in water.

This standard applies to 1-naphthol (alpha-naphthol) and 2- in surface water, groundwater, domestic sewage, industrial wastewater and seawater.

Determination of naphthol (beta-naphthol).

When the sampling volume is 10.0 ml, the eluent volume is 10.0 ml, and the injection volume is 10.0 μ l, the emission wavelength is 425 nm.

Here, the detection limits of the 1-naphthol and 2-naphthol methods of this standard are 2 μ g/L, and the lower detection limits are 8 μ g/L.

At 360 nm, the detection limit of 2-naphthol by this standard is 0.2 μ g/L, and the lower detection limit is 0.8 μ g/L.

When the sampling volume is 50.0 ml, the eluent volume is 10.0 ml, and the injection volume is 10.0 μ l, the emission wavelength is 425 nm.

Here, the detection limits of the 1-naphthol and 2-naphthol methods of this standard are both 0.3 μ g/L, and the lower detection limits are 1.2 μ g/L.

At the emission wavelength of 360 nm, the detection limit of the method for the determination of 2-naphthol by this standard is 0.06 μ g/L, and the lower detection limit is 0.24 μ g/L.

2 Normative references

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

GB 17378.3 Marine Monitoring Code Part 3. Sample Collection, Storage and Transportation
HJ 91.1 Technical Specifications for Sewage Monitoring HJ/T 91 Technical specifications for surface water and sewage monitoring HJ/T 164 Technical Specifications for Groundwater Environmental Monitoring

3 Method principle

Take a certain amount of acidified sample (pH value 1 ~ 2), extract it with a C18 solid phase extraction column, and elute with methanol.

The naphthol was separated by liquid chromatography and detected by a fluorescence detector. Qualitative according to retention time, quantified by external standard method.

4 Interference and cancellation

4.1

Basic organic compounds such as naphthylamine and aniline in water will not be solidified by C18 under acidic conditions (pH 1-2) Adsorption by extraction column does not interfere with the determination of naphthol.

4.2

Common phenolic compounds (including phenol, 2,4-dinitrophenol, 2,6-dimethylphenol, 2-methylphenol, 3-methylphenol, 4-methylphenol, 4-chlorophenol, 2,4-dichlorophenol, 2,4,6-trinitrophenol, 1,3-benzenediol, p-nitrophenol, o-chlorophenol, o-nitro Phenol, 2,4-xyleneol, 4-chloro-m-cresol, 4,6-dinitro-o-cresol, 2,4,6-trichlorophenol and pentachlorophenol, etc.) it does not interfere with the determination of naphthol at a fixed fluorescence detection wavelength.

4.3

Other acidic or neutral organic compounds with similar retention time to naphthol may interfere with the determination of naphthol.

Changing the ratio of the mobile phase improves the resolution, and you can choose to measure at a relatively interference-free detection wavelength to avoid interference.

5 Reagents and materials

Unless otherwise stated, analytical reagents that meet national standards are used in the analysis. The test water is free of target compounds.

Distilled water or water produced by pure water equipment.

5.1

Acetonitrile (CH_3CN). pure by liquid chromatography.

5.2

Methanol (CH_3OH). pure by liquid chromatography.

5.3

Sodium hydroxide (NaOH).

5.4

Ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$).

5.5 1-naphthol. w ($\text{C}_{10}\text{H}_7\text{OH}$) $\geq 99.5\%$.

5.6 2-naphthol. w ($\text{C}_{10}\text{H}_7\text{OH}$) $\geq 99.5\%$.

5.7

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

5.8

hydrochloric acid solution. 1 1.

5.9

Hydrochloric acid solution. $c(\text{HCl}) = 0.01 \text{ mol/L}$.

Measure 1.8 ml of hydrochloric acid solution (5.7) and dilute to 1000 ml with water.

5.10

Sodium hydroxide solution. $c(\text{NaOH}) = 5 \text{ mol/L}$.

Weigh 20 g of sodium hydroxide (5.3) and dissolve in 100 ml of water.

5.11

Methanol solution. w (CH_3OH) = 0.2%.

Take 2.0 ml of methanol (5.2) and dilute to 1000 ml with water.

5.12

Naphthol mixed standard stock solution. $\rho \sim 1000 \text{ mg/L}$.

Weigh 0.10 g (accurate to 0.0001 g) of 1-naphthol (5.5) and 2-naphthol (5.6) respectively in a 100 ml beaker, Add 100 mg ascorbic acid (5.4), dissolve with a small amount of methanol (5.2), transfer to a 100 ml volumetric flask, and use methanol (5.2) Dilute to the mark and mix well. Divided into brown sample bottles (6.4), sealed, refrigerated below 4°C , protected from light Save for 3 months. You can also purchase a commercially available