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HJ

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

HJ 1072-2019

**Water quality--Determination of pyridine--Headspace / gas
chromatography**

水质 吡啶的测定 顶空气相色谱法

(English Translation)

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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 pyridine in water, and formulate this standard.

This standard specifies headspace/gas chromatography for the determination of pyridine in surface water, groundwater, domestic sewage and industrial wastewater.

Compared with "Gas Chromatography for the Determination of Water Quality Pyridine" (GB/T 14672-1993), the main differences between this standard are as follows.

- The name was changed to "headspace/gas chromatography for the determination of pyridine in water";
- Added normative references;
- Added description of method principle;
- The chromatographic column was changed from a packed column to a capillary column, and the manual headspace sample was changed to a fully automatic headspace sampler;

- Quantitative by standard curve external standard method;
- Added quality control and quality assurance;
- Added waste disposal and precautions.

Since the date of implementation of this standard, the "Pyridine of Water Quality" approved and issued by the former State Environmental Protection Administration on September 18, 1993 Determination of Gas Chromatography (GB/T 14672-1993) in the corresponding environmental quality standards and pollutant emission (control) standards Execution will stop during the implementation.

Appendix A of this standard is an informative appendix.

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. China Shipbuilding Industry Corporation 718 Research Institute.

This standard is verified by. Hebei Province Environmental Monitoring Center Station, Shijiazhuang City Environmental Monitoring Station, Anyang City Environmental Monitoring Station, Baoding Environmental Monitoring Station, China Shipbuilding Chemicals Testing Center and Beijing Huaxue North Testing Technology Co., 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 pyridine--Headspace/gas chromatography Warning. The solvents and standard samples used in the experiment are toxic and harmful compounds. Their reagent preparation and sample preparation process it should be carried out in a fume hood, and protective equipment should be worn during operation to avoid contact with skin and clothing.

1 Scope

This standard specifies headspace/gas chromatography for the determination of pyridine in water.

This standard applies to the determination of pyridine in surface water, groundwater, domestic sewage and industrial wastewater.

When the sampling volume is 10.0 ml, the detection limit of the method is 0.03 mg/L, and the lower limit of determination is 0.12 mg/L.

2 Normative references

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

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

Place the sample in a sealed headspace bottle. Under certain conditions, the pyridine in the sample in the headspace bottle volatilizes to the headspace.

Vapor pressure is generated and thermodynamic equilibrium is reached in the gas-liquid two-phase. Take a part of the gas sample and separate it by gas chromatography, hydrogen flame Detection by ionization detector. Qualitative according to retention time, quantified by standard curve external standard method.

4 Reagents and materials

Unless otherwise stated, analytical reagents that comply with national standards are used in the analysis. The experimental water is double distilled water or pure Water prepared by water equipment must be blank checked before use to confirm that it does not contain the target compound and its retention time is within the target compound.

No interfering chromatographic peaks appeared in the interval.

4.1

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

4.2

Sodium hydroxide (NaOH).

4.3

Sodium chloride (NaCl). excellent grade pure.

Burn at 500°C to 550°C for 2 h, cool to room temperature, and store in a desiccator for later use.

4.4

Sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$).

4.5

Methanol (CH_3OH). chromatographically pure.

4.6

Pyridine (C₅H₅N). purity $\geq 99.5\%$.

4.7

Sulfuric acid solution. 1 1.

4.8

Sodium hydroxide solution. $c(\text{NaOH}) = 10 \text{ mol/L}$.

4.9

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

Add a suitable amount of methanol (4.5) to a 100 ml volumetric flask, and then weigh 100 mg (accurate to 0.1 mg) of pyridine (4.6) Add to volumetric flask, make up to volume with methanol (4.5), and mix. Refrigerate below 4 ° C, sealed and protected from light, can be stored 3 months, return to room temperature before use. Or purchase a certified standard solution directly.

4.10

Standard use solution. $\rho = 10.0 \text{ mg/L}$.

Dilute the standard stock solution (4.9) with water and prepare immediately before use.

4.11

Carrier gas. High-purity nitrogen with a purity of $\geq 99.999\%$.

4.12

Combustion gas. high-purity hydrogen, with a purity of $\geq 99.999\%$.

4.13

Gas-assisting gas. air, dehydrated by silica gel, and activated carbon to remove organic matter.

5 Instruments and equipment

5.1

Sampling bottle. a 40 ml brown screw-top glass bottle with a silicone rubber-teflon liner screw cap.

5.2

Gas chromatograph. with split/splitless inlet and hydrogen flame ionization detector (FID), chromatographic data processing tool Work stop.