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HJ

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

HJ 825-2017

**Water quality-Determination of volatile phenols - Flow injection
analysis(FIA) and 4-AAP spectrophotometric method**

水质 挥发酚的测定 流动注射4-氨基安替比林分光光度法

(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 the Prevention and Control of Water Pollution, Protect the human health, regulate the determination of volatile phenol in water, the development of this standard.

This standard specifies the determination of volatile water in surface water, groundwater, domestic sewage and industrial wastewater by injection of 4-amino Antipyrine spectrophotometric method.

This standard is the first release.

This standard is organized by the Environmental Monitoring Department of the Ministry of Environmental Protection, the Secretary for Science and Technology Standards.

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

The standard verification unit. Beijing Environmental Protection Monitoring Center, Wuxi City Environmental Monitoring Center Station, Huai'an City Environmental Monitoring Central station, Yangzhou City Environmental Monitoring Center Station, Changshu City

Environmental Monitoring Station and Jiangsu Province Environmental Monitoring Center.

The environmental protection department of this standard approved on March 30, 2017.

This standard has been implemented since May 1, 2017.

This standard is explained by the Ministry of Environmental Protection.

Determination of Volatile Phenol in Water by Flow Injection - 4 - Aminoantipyrine Spectrophotometric Method Warning. Phenols are highly toxic substances, the operation should be required to wear protective equipment, to avoid contact with skin, after testing Residual residue should be properly handled safely.

1 Scope of application

This standard specifies the flow injection of 4-aminoantipyrine spectrophotometric method for the determination of volatile phenols in water.

This standard is applicable to the determination of volatile phenol in surface water, groundwater, domestic sewage and industrial waste water.

When the detection range of 10 mm, the standard method of detection limit of 0.002 mg/L, the determination range of 0.008 mg/L ~ 0.200 mg/L.

2 Normative reference documents

The contents of this standard refer to the following documents or their terms. Those who do not specify the date of the reference file, the effective version of the appropriate for this standard.

Determination of Volatile Phenol in Water Quality 4 - Amino Antipyrine Spectrophotometric Method Technical specification for surface water and wastewater monitoring Technical specification for groundwater environmental monitoring

3 Terms and definitions

The following terms and definitions apply to this standard.

Volatile phenols

With the steam distillation and can react with 4-aminoantipyrine to produce colored compounds of volatile phenolic compounds, knot Fruit to phenol.

4.1 Flow injection device works

In a closed line, a volume of the sample into the continuous flow of the carrier liquid, the sample and reagents in the chemical reaction mode the blocks are mixed and reacted in a specific order and proportion, and the reaction is carried out in a non-complete reaction

condition into the flow detection cell for photometric inspection Measurement.

4.2 Principles of chemical reactions

Under acidic conditions, the sample was released from the column by $160^{\circ}\text{C} \pm 2^{\circ}\text{C}$. The distorted phenolic compounds are weak Alkaline medium, in the presence of potassium ferricyanide, and 4-amino antipyrine reaction to produce orange-yellow antipyrine dyes, in Absorbance was measured at 500 nm wavelength. Specific work flow shown in Figure 1.

1. Peristaltic pump; Heating pool (160°C); Phase separation device; Condenser; Injection valve; Reaction ring; Detection pool 10mm, 100 nm; Phosphoric acid solution (6.18); Potassium ferricyanide buffer (6.21); R3.4-aminoantipyrine solution (6.20); Contained Liquid (experimental water); Ice water ($0-4^{\circ}\text{C}$); Sample; Waste liquid

Figure 1 flow injection-4-amino antipyrine spectrophotometric determination of volatile phenol reference work flow chart

5 Interference and elimination

The main disturbances of this method are oxidants, oils, sulfides, organic or inorganic reducing substances and anilines.

5.1 Elimination of oxidizing agents such as free chlorine

The sample drops on the starch-potassium iodide test paper and appears blue, indicating the presence of an oxidizing agent, which can be added to excess iron sulfate to remove it.

5.2 Elimination of sulfide

When the sample has a black precipitate, it is desirable to take a drop of sample on the lead acetate test paper, if the test paper turns black, indicating sulfur the presence of chemicals. At this point the sample continued to add phosphoric acid acidification, set the ventilation cabinet to stir aeration until the formation of hydrogen sulfide completely Yi Out

5.3 Elimination of organic or inorganic reducing substances such as formaldehyde and sulfite

Can be divided into appropriate sample in the separatory funnel, add sulfuric acid solution to make acidic, were added 50 ml, 30 ml, 30 ml Ether to extract phenol, combined ether layer in another separatory funnel, by adding 4 ml, 3 ml, 3 ml of sodium hydroxide solution into Line back extraction, so that phenols into sodium hydroxide solution. Combined with alkali extract, into the beaker, set the water bath to heat, in addition to to remove the residual ether, and then use the experimental water to dilute the alkali extract to the original volume of water samples.

At the same time should be experimental water for blank test.

5.4 Elimination of oil

After the sample was left to separate the oil slick, follow the steps in 5.3.

5.5 Elimination of aniline

Aniline can be detected with 4-aminoantipyrine color reaction to interfere with the determination of phenol, generally in the acidic (pH 6 reagents and materials Unless otherwise stated, analytical reagents conforming to national standards are used for analysis. The experimental water is freshly prepared Ionized water or distilled water. In addition to the standard solution, other solutions and experimental water are treated with helium (6.28) or ultrasonic (7.3) degassed.

6.1 Phenol-free water. Prepared according to the method specified in HJ 503.

6.2 Purified phenol (C_6H_5OH). Prepared according to the procedure specified in HJ 503.

6.3 sulfuric acid. $\rho(H_2SO_4) = 1.84 \text{ g/ml}$, excellent grade pure.

6.4 Phosphoric acid. $\rho(H_3PO_4) = 1.69 \text{ g/ml}$, excellent grade pure.

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

6.6 Sodium hydroxide ($NaOH$).

6.7 boric acid (H_3BO_3).

6.8 Potassium ferricyanide [$K_3Fe(CN)_6$].

6.9 Potassium chloride (KCl).

6.10 Potassium Bromate ($KBrO_3$).

6.11 Potassium Bromide (KBr).

6.12 Potassium iodate (KIO_3).

6.13 Sodium thiosulfate ($Na_2S_2O_3$).

6.14 Sodium carbonate (Na_2CO_3).

6.15 Potassium iodide (KI).

6.16 4-aminoantipyrine ($C_{11}H_{13}N_3O$).

6.17 sulfuric acid solution. 1 5.

The volume ratio of sulfuric acid (6.3) to water is 1. 5.

6.18 Phosphoric acid solution. 1 10.