

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

HJ 824-2017

**Water quality - Determination of sulfide-Flow injection analysis
(FIA) and methylene blue spectrophotometric method**

水质 硫化物的测定 流动注射-亚甲基蓝分光光度法

(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, Protection of human health, regulate the determination of sulfide in water methods, the development of this standard.

This standard specifies the determination of surface water, groundwater, domestic sewage and industrial waste water sulfide flow injection - methylene blue Spectrophotometry.

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. Nanjing Environmental Monitoring Center Station, Huai'an City Environmental Monitoring Center Station, Yangzhou City Environmental Monitoring Heart station, Changshu City Environmental Monitoring Station, Jiangsu Province, Disease Prevention and Control Center 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.

Water quality - Determination of sulfide - Flow injection - methylene blue spectrophotometric method

1 Scope of application

This standard specifies the flow injection-methylene blue spectrophotometric method for the determination of sulfides in water.

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

When the optical path length is 10 mm, the detection limit of this method is 0.004 mg/L (in S2), the determination range is 0.016 Mg/L 2.00 mg/L (in S2).

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.

Water quality - Determination of sulfide - Methylene blue spectrophotometric method GB/T 16489 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.

Sulfide

Refers to the sum of dissolved inorganic sulfides and acid-soluble metal sulfides in water. Including the solubility of H₂S, HS⁻, S₂⁻, and storage in the suspended matter in the soluble sulfide and acid soluble metal sulfide.

4.1 Flow injection analyzer works

In a closed line, a volume of the sample is injected into the continuous flow of the carrier liquid, the sample and reagent in the chemical reaction module Specific order and ratio of mixing, reaction, in the non-complete reaction conditions, into the flow detection pool for photometric detection.

4.2 Principles of chemical reactions

In the acidic medium, the sample through $65\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ on-line heating release of hydrogen sulfide gas is absorbed by sodium hydroxide solution. Absorbent liquid Sulfide reacts with p-aminodimethylaniline and ferric chloride to form methylene blue, and absorbance is measured at 660 nm. Specific work the process is shown in Figure 1.

1. Peristaltic pump; Heating pool ($65\text{ }^{\circ}\text{C}$); Diffusion pool; Injection valve; Reaction ring; Heating pool ($30\text{ }^{\circ}\text{C}$); Detection pool 10mm, 660nm;

R1 Phosphoric acid solution (6.20); Sodium hydroxide solution (6.23); R3. Picoline dimethylaniline solution (6.25); R4 Ferric chloride solution (6.24);

C. Carrier solution (sodium hydroxide solution, 6.23); Sample; Waste liquid

Figure 1 flow injection - spectrophotometric determination of sulfide reference work flow chart

5 Interference

The main disturbances of this method are SO_3^{2-} , $\text{S}_2\text{O}_3^{2-}$, SCN^- , NO_2^- , CN^- , Cu^{2+} , Pb^{2+} , Hg^{2+} . The sulfide content was 0.50 mg/L, The maximum permissible levels of interfering substances in the samples were 20 mg/L SO_3^{2-} , 240 mg/L $\text{S}_2\text{O}_3^{2-}$, 400 mg/L SCN^- , 65 mg/L NO_2^- , 200 mg/L NO_3^- , 400 mg/L I^- , 5 mg/L CN^- , 2 mg/L Cu^{2+} , 25 mg/L Pb^{2+} and 4 mg/L Hg^{2+} .

6 Reagents and materials

Unless otherwise stated, analytical reagents conforming to national standards are used for analysis. The experimental water is freshly prepared for deionized water or Distilled water. In addition to the standard solution, other solutions and experimental water are treated with helium (6.33) or ultrasonic (7.3) degassed.

6.1

hydrochloric acid. $\rho(\text{HCl}) = 1.19\text{ g/ml}$, excellent grade pure.

6.2

Phosphoric acid. $\rho(\text{H}_3\text{PO}_4) = 1.69\text{ g/ml}$, excellent grade pure.

6.3

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

6.4

Sodium hydroxide (NaOH). excellent grade pure.

6.5

Ferric trichloride [$\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$].

6.6

p-aminodimethylaniline [$(\text{CH}_3)_2\text{NC}_6\text{H}_4\text{NH}_2 \cdot 2\text{HCl}$].

6.7

Sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$).

6.8

Sodium sulfide ($\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$).

6.9

Sodium carbonate (Na_2CO_3).

6.10

Potassium iodide (KI).

6.11

Zinc acetate ($\text{ZnAc}_2 \cdot 2\text{H}_2\text{O}$).

6.12

Sodium acetate ($\text{NaAc} \cdot 3\text{H}_2\text{O}$).

6.13

Iodine (I_2).

6.14

Starch ($\text{C}_6\text{H}_{10}\text{O}_5$) n.

6.15

potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$). standard or excellent grade pure.

After drying at 105 °C for 2h, stand-by.

6.16

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

6.17

sulfuric acid solution. 1 5.

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

6.18

hydrochloric acid solution. c (HCl) = 3 mol/L.