

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 1226-2021

**Water quality - Determination of sulfide - Methylene blue
spectrophotometric method**

水质 硫化物的测定 亚甲基蓝分光光度法

(English Translation)

Issued on: January 1, 1970

Implemented on: January 1, 1970

Issued by: Ministry of Ecology and Environment

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1 Scope of application

This standard specifies the 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, industrial wastewater and seawater.

When the sampling volume is 200 ml and a 10 mm optical path cuvette is used, the detection limit of the method is 0.01 mg/L, and the lower detection limit is 0.04 mg/L;

When using a 30 mm optical path cuvette, the detection limit of the method was 0.003 mg/L, and the lower limit of determination was 0.012 mg/L.

Note: The 30 mm optical path cuvettes are only used for the determination of groundwater or low-concentration seawater below the first class standard, and the pretreatment method should use "acidification-distillation-absorption" Law.

2 Normative references

This standard refers to the following documents or clauses thereof. For dated references, only the dated version applies to this standard.

For undated references, the latest edition (including all amendments) applies to this standard.

HJ/T 91 Technical Specification for Surface Water and Sewage Monitoring HJ 91.1
Technical Specification for Wastewater Monitoring HJ 164 Technical Specification for
Groundwater Environmental Monitoring HJ 442.3 Technical Specifications for
Environmental Monitoring in Offshore Seas Part III Water Quality Monitoring in Offshore
Seas HJ 493 Technical regulations on preservation and management of water sampling
samples

3 Terms and Definitions

The following terms and definitions apply to this standard.

sulfide

Water-soluble inorganic sulfides and acid-soluble metal sulfides, including soluble H_2S , $-\text{HS}$, $2-\text{S}$, and in suspensions of soluble sulfides and acid-soluble metal sulfides.

4 Principles of the method

After the sulfide in the sample is acidified, heated with nitrogen blowing or distilled, the generated hydrogen sulfide is absorbed by sodium hydroxide solution, and the generated sulfide ions are it reacts with N,N-dimethyl-p-phenylenediamine in an acidic solution of ferric ammonium sulfate to generate methylene blue, and its absorbance is measured at 665 nm.

The chemical content is proportional to the absorbance value.

5 Interference and cancellation

The main interferent is 2SO_3^{2-} , 2SO_3^{2-} , SCN^- , NO_3^- , I^- , NO_2^- , CN^- and some heavy metal ions. Sulfide content is 0.3 mg/L, the maximum permissible content of interfering substances in the sample is 2SO_3^{2-} - 700 mg/L, 2SO_3^{2-} - 900 mg/L, SCN^- - 900 mg/L, NO_3^- - 200 mg/L, - 400 mg/L, CN^- - 5 mg/L, 2Cu 2 mg/L, 2Pb 25 mg/L, 2Hg 4 mg/L. NO_2^- - Can react with methylene blue to enable assay the result is low, NO_2^- - This method is not applicable when the concentration (in N) is higher than 2.0 mg/L.

6 Reagents and Materials

Unless otherwise stated, analytical reagents that meet national standards were used in the analysis, and the experimental water was freshly prepared deionized water or distilled water.

6.1

De-oxygenated deionized water. Deionized water is prepared through an ion exchange column, and nitrogen is passed through at a rate of 200 ml/min to 300 ml/min for about 20 minutes.

Saturate the water with nitrogen to remove dissolved oxygen from the water. The prepared deoxygenated deionized water should be immediately sealed and stored in a glass bottle. Instant use.

6.2

Sulfuric acid (H_2SO_4). $\rho=1.84$ g/ml.

6.3

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

6.4

Sodium Hydroxide (NaOH).

6.5

N,N-Dimethyl-p-phenylenediamine hydrochloride $[\text{NH}_2\text{C}_6\text{H}_4\text{N}(\text{CH}_3)_2 \cdot 2\text{HCl}]$.

6.6

Ferric ammonium sulfate $[\text{Fe}(\text{NH}_4)(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$.

6.7

Zinc acetate $[\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}]$.

6.8

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

6.9

Disodium EDTA ($\text{C}_{10}\text{H}_{14}\text{O}_8\text{N}_2\text{Na}_2 \cdot 2\text{H}_2\text{O}$).

6.10

Hydrochloric acid solution.

Measure 250 ml of hydrochloric acid (6.3) and slowly pour it into 250 ml of water and cool it.

6.11

Zinc acetate solution. $c[\text{Zn}(\text{CH}_3\text{COO})_2]=1 \text{ mol/L}$.

Weigh 220 g of zinc acetate (6.7), dissolve it in 1000 ml of water, and filter it before use if it is cloudy.

6.12

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

Weigh 10.0 g of sodium hydroxide (6.4) and dissolve it in 1000 ml of water, shake well.

6.13

Antioxidant solution.

Weigh 4.0 g ascorbic acid (6.8), 0.2 g disodium EDTA (6.9), 0.6 g sodium hydroxide (6.4) and dissolve in 100 ml water, shake well and store in a brown reagent bottle. Instant use.

6.14

N,N-dimethyl-p-phenylenediamine solution. $\rho[\text{NH}_2\text{C}_6\text{H}_4\text{N}(\text{CH}_3)_2 \cdot 2\text{HCl}]=2 \text{ g/L}$.

Weigh 2.0 g of N,N-dimethyl-p-phenylenediamine hydrochloride (6.5) and dissolve it in 700 ml of water, slowly add 200 ml of sulfuric acid (6.2), and cool. Then dilute to 1000 ml with water and shake well. This solution is stable for 3 months when stored in an airtight amber bottle at room temperature.

6.15

Ferric ammonium sulfate solution. $\rho[\text{Fe}(\text{NH}_4)(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]=100 \text{ g/L}$.

Weigh 25.0 g of ferric ammonium sulfate (6.6) and dissolve it in 100 ml of water, slowly add 5.0 ml of sulfuric acid (6.2), and dilute with water to 250 ml, shake well. If there is insoluble matter in the solution, it should be used after filtration.

6.16

Sulfide standard solution. you can buy commercially available certified reference materials, or you can prepare it yourself. See Appendix A for the preparation and calibration methods.

6.17

Sulfide standard solution. $\rho(\text{S}^{2-})=10.00 \text{ mg/L}$.

Transfer a certain amount of sulfide standard solution (6.16) to 2.0 ml of sodium hydroxide solution (6.12) and an appropriate amount of deionized water.

(6.1) in a 100 ml brown volumetric flask, dilute to volume with deoxygenated deionized water (6.1), and prepare a sulfur containing sulfur ion concentration of 10.00 mg/L.

Chemical standard solution. Instant use.