

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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Water quality Determination of azide spectrophotometric method

水质 叠氮化物的测定 分光光度法

(English Translation)

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1 Scope of application

This standard specifies a spectrophotometric method for the determination of azides in water.

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

When the sampling volume is 150 ml, the sample preparation volume is 100 ml, and the 10 mm pathlength cuvette is used, the detection limit of the method is 0.08 mg/L (calculated as azide root), the lower limit of determination is 0.32 mg/L (calculated as azide root).

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

3 Terms and Definitions

The following terms and definitions apply to this standard.

azide

Under the conditions specified in this standard, azide compounds of azide acid can be formed by heating and distillation, calculated as azide radicals.

4 Principles of the method

The azide in the sample is heated and converted into azide acid in an acidic medium, which is distilled out with water vapor and absorbed by sodium hydroxide solution.

The root ion reacts with ferric ion to form a brown-red complex, and its absorbance is measured at 454 nm. Within a certain concentration range, its absorption the luminosity is proportional to the azide ion content.

5 Interference and cancellation

5.1

The interference of chromaticity, CN^- , SCN^- , sulfide and common metal ions in the sample can be effectively removed by heating distillation.

5.2 Presence of NO_2 in the sample

-, it interferes with the determination of azide, which can be eliminated by adding ammonium sulfamate before distillation. 5.0 ml Ammonium Sulfamate the solution (6.12) can eliminate 25.0 mg/L of NO_2 -interference.

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

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

6.2

Sodium Hydroxide (NaOH).

6.3

Ferric perchlorate $[\text{Fe}(\text{ClO}_4)_3 \cdot \text{H}_2\text{O}]$. chemically pure.

6.4

Sodium azide. $w(\text{NaN}_3) \geq 99.5\%$.

6.5

Ammonium sulfamate $(\text{NH}_4\text{SO}_3\text{NH}_2)$.

6.6

Sodium hydroxide solution. $\rho(\text{NaOH}) = 40 \text{ g/L}$.

Weigh 20.00 g of sodium hydroxide (6.2), dissolve with a small amount of water, dilute to 500 ml, and store in a polyethylene bottle.

6.7

Sodium hydroxide solution. $\rho(\text{NaOH}) = 400 \text{ mg/L}$.

Weigh 0.40 g of sodium hydroxide (6.2), dissolve it with a small amount of water, dilute it to 1000 ml, and store it in a polyethylene bottle.

6.8

Sodium hydroxide solution. $\rho(\text{NaOH}) = 40 \text{ mg/L}$.

Measure 100 ml of sodium hydroxide solution (6.7), dilute to 1000 ml with water, and store in a polyethylene bottle.

6.9

Hydrochloric acid solution. $c(\text{HCl}) = 1.0 \text{ mol/L}$.

Measure 9.0 ml of hydrochloric acid (6.1), dilute it with water to 100 ml, and store it in a ground glass bottle.

6.10

Hydrochloric acid solution. $c(\text{HCl}) = 0.1 \text{ mol/L}$.

Measure 10.0 ml of hydrochloric acid solution (6.9), dilute to 100 ml with water, and store in a ground-mouth glass bottle.

6.11

Ferric perchlorate solution. $\rho[\text{Fe}(\text{ClO}_4)_3 \cdot \text{H}_2\text{O}] = 50 \text{ g/L}$.

Weigh 5.00 g of ferric perchlorate (6.3), dissolve with 20 ml of hydrochloric acid solution (6.10), transfer to a 100 ml volumetric flask and dilute with water. Make up to the mark, shake well, and store in a brown ground glass bottle. Refrigerated below 4 °C and protected from light can be stored for 3 months.

6.12

Ammonium sulfamate solution. $\rho(\text{NH}_4\text{SO}_3\text{NH}_2)=150 \text{ g/L}$.

Weigh 15.00 g of ammonium sulfamate (6.5), dissolve it in a small amount of water, transfer it to a 100 ml volumetric flask, dilute it to the mark, and shake well.

Ready to use.

6.13 Standard stock solution of sodium azide. $\rho(\text{N}_3)$

$\rho(\text{N}_3)=1.00 \text{ g/L}$.

Accurately weigh 0.1548 g of sodium azide (6.4), dissolve it with a small amount of sodium hydroxide solution (6.8), transfer it to a 100 ml volumetric flask and Dilute to the mark with sodium hydroxide solution (6.8), shake well, and store in a polyethylene bottle. Refrigerated below 4 °C and protected from light can be stored for 3 months.

6.14 Standard solution of sodium azide. $\rho(\text{N}_3)$

$\rho(\text{N}_3)=25.0 \text{ mg/L}$.

Pipette 2.50 ml sodium azide standard stock solution (6.13) into a 100 ml volumetric flask, dilute it with sodium hydroxide solution (6.8) to Line, shake. Ready to use.

6.15 Glass beads. 4 mm to 6 mm in diameter.

7 Instruments and equipment

Unless otherwise stated, the A-grade glass measuring apparatus in accordance with national standards was used in the analysis.

7.1

Sampling bottle. 500 ml amber glass bottle.

7.2

Spectrophotometer. equipped with 10 mm optical path cuvettes.

7.3

Electric furnace. power 0 W ~ 1000 W, adjustable.

7.4

Distillation device. It consists of an all-glass still (with a 500 ml distillation flask and a condenser), a receiving flask and a distillate conduit.

7.5

Receiving bottle. 100 ml colorimetric tube with stopper.