

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 665-2013

**Water quality. Determination of ammonium nitrogen by
continuous flow analysis (CFA) and Salicylic acid
spectrophotometry**

水质 氨氮的测定 连续流动-水杨酸分光光度法

(English Translation)

Issued on: January 1, 1970

Implemented on: January 1, 1970

Issued by: Ministry of Ecology and Environment

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5.13 Sulfuric acid solution I. $c(\text{H}_2\text{SO}_4)=0.16\text{mol/L}$

5.14 Sulfuric acid solution II. $c(\text{H}_2\text{SO}_4)=0.5\text{mol/L}$

5.15 Sodium hydroxide solution. $\rho(\text{NaOH})=0.2\text{g/ml}$

5.17 Dodecyl polyglycol ether (Brij35) solution. $\omega=30\%$

5.18 Buffer Solution I. ($\text{pH}=5.2$)

5.19 Buffer Solution II. ($\text{pH}=5.2$)

5.22 Sodium nitroferricyanide solution. $\omega(\text{Na}_2[\text{Fe}(\text{CN})_5\text{NO}]\cdot 2\text{H}_2\text{O})=0.1\%$

5.23 Sodium dichloroisocyanurate solution I. $\omega(\text{C}_3\text{Cl}_2\text{N}_3\text{O}_3\text{Na}\cdot 2\text{H}_2\text{O})=0.2\%$

5.24 Sodium dichloroisocyanurate solution II. $\omega(\text{C}_3\text{Cl}_2\text{N}_3\text{O}_3\text{Na}\cdot 2\text{H}_2\text{O})=3.5\%$

5.25 Ammonia nitrogen standard stock solution. $\rho(\text{N})=1000\text{mg/L}$

5.26 Ammonia nitrogen standard use solution I. $\rho(\text{N})=100\text{mg/L}$

5.27 Ammonia nitrogen standard use solution II. $\rho(\text{N})=10.0\text{mg/L}$

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5.30 Nitrogen. purity $\geq 99\%$

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Foreword

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 Prevention and Control, to protect the environment, to ensure human health, standardize the determination method of ammonia nitrogen in water, and formulate this standard.

This standard specifies a continuous flow-salicylic acid spectrophotometric method for the determination of ammonia nitrogen in water.

This standard is the first release.

This standard was formulated by the Science and Technology Standards Department of the Ministry of Environmental Protection.

This standard is mainly drafted by. Suzhou Environmental Monitoring Center Station.

This standard is verified by. Jiangyin Environmental Monitoring Station, Taihu Basin Water Environment Monitoring Center, and Jilin City Environmental Protection Monitoring Station, Tianjin Water Environment Monitoring Center, Yellow River Basin Water Environment Monitoring Center and Suzhou Environmental Monitoring Center Station.

This standard was approved by the Ministry of Environmental Protection on October 25, 2013.

This standard has been implemented since January 1, 2014.

This standard is explained by the Ministry of Environmental Protection.

Determination of ammonia nitrogen in water - continuous flow - salicylic acid spectrophotometric method

1 Scope of application

This standard specifies a continuous flow-salicylic acid spectrophotometric method for the determination of ammonia nitrogen in water.

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

When the direct colorimetric module is used and the detection cell optical path is 30 mm, the detection limit of the method is 0.01 mg/L (in terms of N).

The measurement range is from 0.04 mg/L to 1.00 mg/L; when the on-line distillation module is used, the detection method is performed when the optical path of the detection cell is 10 mm.

The limit is 0.04 mg/L (in terms of N), and the measurement range is from 0.16 mg/L to 10.0 mg/L.

2 Normative references

The contents of this standard refer to the terms in the following documents. For undated references, the valid version applies to this standard.

HJ 536 Water quality - Determination of ammonia nitrogen - Salicylic acid spectrophotometric method HJ/T 91 Surface Water and Wastewater Monitoring Technical Specifications HJ/T 164 Technical Specifications for Groundwater Environmental Monitoring

3.1 Working principle of continuous flow analyzer

The sample and the reagent enter the chemical reaction module under the push of the peristaltic pump, and continuously flow in the closed pipeline, and are pressed by the bubble.

They are regularly spaced at regular intervals and mixed and reacted in a specific order and proportion. After the color development is completed, they enter the flow detection cell.

Photometric detection.

3.2 Principles of chemical reactions

In an alkaline medium, the ammonia and ammonium ions in the sample react with the hypochlorite released from the sodium dichloroisocyanurate solution.

Chloramine. Chloramine reacts with salicylate to form a blue-green compound in the presence of 40 ° C and potassium nitroferrocyanide.

The absorbance was measured at a wavelength of 660 nm.

Refer to the workflow diagram, see Figure 1 and Figure 2.

1 peristaltic pump 2 mixing ring 3 mixing reaction ring 4 heating pool (circle) 40 ° C G air

5 Flow detection cell 30mm 660nm 6 De-bubble S sample 0.60ml/min W Waste liquid

R1 buffer solution 1 (5.18) 0.60ml/min R2 sodium salicylate solution I (5.20) 0.32ml/min R3 sodium nitroferricyanide solution (5.22) 0.16ml/min R4 sodium dichloroisocyanurate solution I (5.23) 0.32ml/min

Figure 1 Flow chart of direct reference colorimetric determination of ammonia nitrogen

1 peristaltic pump 2 mixing (reaction) ring 3.1 heating bath 120 ° C 3.2 distillation unit 4 heating bath 40 ° C 5 flow detection tank 10mm 660nm 6 degassing W waste liquid R1 Distillation Reagent (5.16) 1.60 ml/min R2 Sulfuric Acid Solution I (5.13) 0.42 ml/min S Sample 0.60 ml/min R3 Buffer Solution II (5.19) 0.80 ml/min R4 Sodium Salicylate Solution II (5.21) 0.32 ml/min G1 Air R5 Sodium dichloroisocyanurate solution II (5.24) 0.23 ml/min ReS Secondary injection 0.16 ml/min G2 Nitrogen (5.30)

Figure 2 Flow chart of the determination of ammonia nitrogen by colorimetry after distillation

4 interference and elimination

4.1 The residual chlorine in the sample will form a chloramine interference assay, which can be removed by adding an appropriate amount of sodium thiosulfate solution (5.28).

4.2 When the concentration of calcium, manganese and chloride ions in the sample is greater than 150mg/L, 10mg/L and 10000mg/L, respectively, Will cause positive interference to the analysis. Samples can be pre-distilled with HJ 536 or directly with modules with in-line distillation (figure

2) Analysis. When the concentration of magnesium ion and iron ion in the sample is not higher than 300 mg/L, there is no influence on the determination of ammonia nitrogen.

4.3 When the pH of the sample is >10 or pH5 reagents and materials Analytically pure reagents in accordance with national standards were used for analysis, unless otherwise stated.

Ammonia-free water with a rate greater than 10 Mohm·cm (25 ° C).

5.1

Hydrochloric acid. $\rho(\text{HCl}) = 1.18\text{g/ml}$.