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HJ

National Environmental Protection Standard of the People's Republic of China

HJ 536-2009

Replaces GB 7481-87

Water quality - Determination of ammonia nitrogen - Salicylic acid spectrophotometry

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Notice

of Ministry of Environmental Protection

of the People's Republic of China

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In order to implement the *Environmental Protection Act of the People's Republic of China*, protect the environment and guarantee human health, hereby we approved and released the *Air and exhaust gas - Determination of ammonia - Nessler's reagent spectrophotometry* and other five standards as national environmental protection standards.

Standard names and standard numbers are as follows:

1. *Ambient air and exhaust gas - Determination of ammonia - Nessler's reagent spectrophotometry* (HJ 533-2009);
2. *Ambient air - Determination of ammonia - Sodium hypochlorite - salicylic acid spectrophotometry* (HJ 534-2009);
3. *Water quality - Determination of ammonia nitrogen - Nessler's reagent spectrophotometry* (HJ 535-2009);
4. *Water quality - Determination of ammonia nitrogen - salicylic acid spectrophotometry* (HJ 536-2009);
5. *Water quality - Determination of ammonium nitrogen - Distillation-neutralization titration* (HJ 537-2009).

The above standards have been implemented since April 1, 2010. They are published by China Environmental Science Press. The standards' content can be found at the website of the Ministry of Environmental Protection (bz.mep.gov.cn).

From the date of the implementation of the above standards, the national environmental standards approved and published by the former State Environmental Protection Administration shall be abolished. The standard names, numbers are as follows:

1. *Air quality - Determination of ammonia - Nessler's reagent colorimetric method* (GB/T 14668-93);
2. *Air quality - Determination of ammonia - Sodium hypochlorite - salicylic acid spectrophotometry* (GB/T 14679-93);
3. *Water quality - Determination of ammonium - Nessler's reagent colorimetric method* (GB 7479-87);
4. *Water quality - Determination of ammonium - salicylic acid spectrophotometry* (GB 7481-87);
5. *Water quality- Determination of ammonium - Distillation and titration method* (GB 7478-87)

Hereby it is noticed.

December 31, 2009

Table of Contents

Foreword 5

1 Application scope 6

2 Method principle 6

3 Interference and elimination 6

4 Reagents and materials 6

5 Instruments and equipment 9

6 Sample 9

7 Analytical procedures 9

8 Results representation 10

9 Accuracy and precision 11

10 Quality assurance and quality control 11

Appendix A 13

Appendix B 15

Foreword

In order to implement the *Environmental Protection Act of the People's Republic of China* and the *Water Pollution Prevention Act of the People's Republic of China*, protect the environment, guarantee human health and standardize the monitoring methods of ammonia nitrogen in water, hereby this standard is announced.

This standard specifies the determination of ammonia nitrogen in water by salicylic acid spectrophotometry.

This standard is revision to *Water quality - Determination of ammonium- salicylic acid spectrophotometry* (GB 7481-87).

This standard was initially published in 1987. And the original drafting organization was Jiangxi Province Ganzhou Environmental Monitor Station. This standard is revised for the first time. The main revised contents are as follows:

— The name of the standard is revised from *Water quality - Determination of ammonium - Salicylic acid Spectrophotometry TO Water quality - Determination of ammonium nitrogen - Salicylic acid spectrophotometry*.

— Add the 30mm cuvette test method. Reduce the detection limits. Broaden the application range of the method. Clearly specify the lower and upper limits of the method.

— Combine the result calculation formulae.

— Modify the normative appendix.

The *Water quality-Determination of ammonium-Salicylic acid Spectrophotometry* (GB 7481-87) approved and released by the former State Environmental Protection Agency on March 14, 1987 shall be abolished, from the date of implementation of this standard.

Appendix A of this standard is normative. Appendix B is informative.

This standard was organized and formulated by Department of Science, Technology and Standards, Ministry of Environmental Protection of the People's Republic of China.

This standard was mainly drafted by: Municipal Environmental Monitoring Center Station of Shenyang.

This standard was approved by Ministry of Environmental Protection on December 31, 2009.

This standard shall be implemented from April 1, 2010.

The Ministry of Environmental Protection is responsible for the interpretation of this standard.

Water quality-Determination of ammonia nitrogen-Salicylic acid spectrophotometry

1 Application scope

This standard specifies the salicylic acid spectrophotometry to determine ammonia nitrogen in water.

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

When the sample volume is 8.0 ml and the 10 mm cuvette is used, the detection limit of this method is 0.01 mg/L, the determination lower limit is 0.04mg/L, and the determination upper limit is1.0mg/L (all counted in N).

When the sample volume is 8.0 ml and the 30mm cuvette is used, the detection limit of this method is 0.004 mg/L, the determination lower limit is 0.016mg/L, and the determination upper limit is 0.25mg/L (all counted in N).

2 Method principle

Under the condition that the alkaline medium (pH=11.7) and the sodium nitroprusside exist, chemical reactions happen AMONG the ammonia, ammonium ions AND the salicylate AND hypochlorite ions in water, to generate blue compound. Measure the absorbency with a spectrophotometer at 697nm.

3 Interference and elimination

This method applies to the possible interfering substance and quantity limit during water sample analysis, see Appendix B for details.

Severe interferences generated by aniline and ethanol amine are rare. Usually the interference is generated by primary amine. When it contains chloramines, excessively high acidity, alkalinity, or when it contains substances that make hypochlorite ions reduction, the interferences may be generated.

If the color of the water sample is too dark, or the salt is excessive, or when the masking ability of the potassium tartrate to the metal ions in the water sample is insufficient, or when there is high concentration of calcium, magnesium and chloride in the water sample, pre-distillation is required.

4 Reagents and materials

Unless otherwise specified, the reagents used in analysis shall be analytical pure

hydroxide solution (4.6), until the salicylic acid is fully dissolved. And use 1mol/L of sulfuric acid to adjust PH of the solution to 6.0-6.5.

Appendix A

(Normative)

The preparation method of sodium hypochlorite solution and calibration of effective chlorine concentration and free-base concentration

A.1 The preparation method of sodium hypochlorite solution

Put hydrochloric (ρ=1.19g/ml) acid, drop by drop, on the solid of potassium permanganate. Guide the escaping chlorine into 2mol/L of sodium hydroxide absorption liquid for absorption, so as to generate the sodium hypochlorite solution in light grass green. Store in a plastic bottle. As this solution is not stable, the effective chlorine concentration shall be calibrated prior to use.

A.2 The measurement of effective chlorine in the sodium hypochlorite solution

Absorb 10.0ml of sodium hypochlorite (4.8) into 100mml volumetric flash. Add water to dilute to marked line. Mix well. Move 10.0ml of diluted sodium hypochlorite solution into 250ml iodine flask. Add 40ml of distilled water and 2.0g potassium iodide. Mix well. Add 5.0 mL of 6mol/L sulfuric acid solution. Mix well. Place in dark for 5min. Use 0.1mol/L of sodium thiosulfate solution to titrate to light yellow. Add approximately 1ml of starch indicator. Continue to titrate it until the blue color disappears. Its mass concentration of effective chlorine shall be calculated as per Equation (A.1):

$$\text{Effective chlorine (g/L, in } Cl_2) = \frac{c \times V \times 35.45}{10.0} \times \frac{100}{10} \qquad (A.1)$$

Where:

c — Sodium thiosulphate solution concentration, mol/L;

V — The volume of consumed sodium thiosulfate at titration, ml;

35.45 — Molar mass of effective chlorine ($1/2Cl_2$), g/mol.

A.3 Determination of free-base (NaOH) in sodium hypochlorite solution

A.3.1 Calibration of hydrochloric acid

Sodium carbonate standard solution: $c(1/2Na_2CO_3)$=0.1000 mol/L. Weigh 2.6500g of anhydrous sodium carbonate that has been dried under 180°C for 2h. Dissolve in cool water this is newly boiled. Move into 50mml volumetric flask. Dilute to marked line.

Methyl orange indicator: ρ =0.5 g/L. Weigh 50mg of methyl red. Dissolve it in 100ml of ethyl alcohol (4.2).

Hydrochloric acid standard titration solution: c(HCl) =0.10mol/L. Weigh 8.5ml of

Appendix B

(Informative)

The influence of co-existing ion and its elimination

Verified by experiments, the tartrate and citrate can be used as the masking agents. Tartrate is used as the masking agent in this standard. When measuring 4μg of ammonia nitrogen as per the experimental method, the ion amount listed in the table below does not interfere the experiment.

Coexisting ions	The permitted amount /μg	Coexisting ions	The permitted amount /μg	Coexisting ions	The permitted amount /μg
Calcium (II)	500	Molybdenum (VI)	100	Boron (III)	250
Magnesium (II)	500	Cobalt (II)	50	Sulfate radical	$2x10^4$
Aluminum (III)	50	Nickel (II)	1000	Phosphate radical	500
Manganese (II)	20	Beryllium (II)	100	Nitrate radical	500
Cuprum (II)	250	Titanium (IV)	20	Nitrite radical	200
Lead (II)	50	Vanadium (V)	500	Fluorinion	500
Zinc (II)	100	Lanthanum (III)	500	Chloride ion	$1x10^5$
Cadmium (II)	50	Cerium (IV)	50	Diphenylamine	50
Iron (III)	250	Gadolinium (III)	500	Triethanolamine	50
Mercury (II)	10	Argentum (I)	50	Aniline	1
Chrome (VI)	200	Antimony (III)	100	Ethanolamine	1
Tungsten (VI)	1000	Tin (IV)	50		
Uranium (VI)	100	Arsenic (III)	100		

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