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NATIONAL ENVIRONMENTAL PROTECTION STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 535-2009

Replacing GB 7479-87

Water Quality – Determination of Ammonia Nitrogen – Nessler's Reagent Spectrophotometry

水质 氨氮的测定

纳氏试剂分光光度法

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Ministry of Environmental Protection of the PRC

Announcement

2009 No.77

To implement *Environmental Protection Law of the People's Republic of China*, protect environment and safeguard the human health, now *Air and Exhaust Gas - Determination of Ammonia - Nessler's Reagent Spectrophotometry* and other four standards are approved to be national environmental protection standards, and are published.

Name and number of the standards are as follows:

- I. *Air and Exhaust Gas - Determination of Ammonia - Nessler's Reagent Spectrophotometry* (HJ 533-2009);
- II. *Ambient Air - Determination of Ammonia - Sodium Hypochlorite - Salicylic Acid Spectrophotometry* (HJ 534-2009);
- III. *Water Quality - Determination of Ammonia Nitrogen – Nessler's Reagent Spectrophotometry* (HJ 535-2009);
- IV. *Water Quality - Determination of Ammonia Nitrogen - Salicylic Acid Spectrophotometry* (HJ 536-2009);
- V. *Water Quality - Determination of Ammonia Nitrogen - Distillation-Neutralization Titration* (HJ 537-2009).

The above standards were implemented since April 1, 2010, published by China Environmental Science Press; the contents of the standards can be checked at the website of the Ministry of Environmental Protection (bz.mep.gov.cn).

Since the implementation date of the above standards, the following five published national environmental protection standards that were approved by the original State Environmental Protection Agency shall be abolished; the name and number of the standards are as follows:

- I. *Air Quality - Determination of Ammonia - Nessler's Reagent Colorimetric Method* (GB/T 14668-93);
- II. *Air Quality - Determination of Ammonia - Sodium Hypochlorite - Salicylic Acid Spectrophotometry* (GB/T 14679-93);
- III. *Water Quality - Determination of Ammonium - Nessler's Reagent Colorimetric*

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Foreword

This Standard was prepared for the purposes of implementing *Environmental Protection Law of the People's Republic of China*, and *Water Pollution Prevention and Control Law of the People's Republic of China*, protecting environment, safeguarding human health, as well as regulating the detection method of ammonia nitrogen in water.

This Standard specified Nessler's Reagent Spectrophotometry to measure the ammonia nitrogen in water.

This Standard revised *Water Quality - Determination of Ammonium - Nessler's Reagent Colorimetric Method* (GB 7479-87).

This Standard was first-time published in 1987; the drafting organization of the original standard was Jiangsu Environmental Monitoring Central Station; this is the first-time revision. The major revised contents are as follows:

- Standard name was changed FROM *Water Quality - Determination of Ammonium - Nessler's Reagent Colorimetric Method* TO *Water Quality - Determination of Ammonia Nitrogen - Nessler's Reagent Spectrophotometry*.
- Add the cuvette's optical path length (10-20mm); reduce the detection limit of the method; expand the applicable scope of the method. Clearly define the upper and lower detection limit of the method.
- Delete the visual colorimetry.
- Regulate and adjust the structure and format of the standard text.
- Add the precautions to the key steps such as main reagent preparation and the sample pretreatment.
- Combine the calculation formula of the results.

Since the implementation date of this Standard, the *Water Quality - Determination of Ammonium - Nessler's Reagent Colorimetric Method* (GB 7479-87) that was approved & published by the original State Environmental Protection Agency on March 14, 1987 shall be abolished.

This Standard was organized-formulated by Science, Technology and Standard Division of Ministry of Environmental Protection, Department.

Chief drafting organization of this Standard: Shenyang Environmental Monitoring Center.

This Standard was approved by Ministry of Environmental Protection on December 31,

Water Quality – Determination of Ammonia Nitrogen – Nessler's Reagent Spectrophotometry

Warning: Mercuric chloride (HgCl_2) and mercuric iodide (HgI_2) are highly toxic substances; avoid the dermal and oral exposure.

1 Scope

This Standard specifies Nessler's reagent spectrophotometry to determine the ammonia nitrogen in water.

This Standard is applicable to determine the ammonia nitrogen in surface water, underground water, domestic sewage, and industrial waste water.

When the volume of water sample is 50ml, and 20mm cuvette is used; the detection limit for this method is 0.025mg/L, the lower determination limit is 0.10mg/L, and the upper determination limit is 2.0mg/L (calculated as per N).

2 Method Principles

Ammonia nitrogen, existed in the free state of ammonia and ammonium ions, reacts with Nessler's reagent, and generates light reddish brown complex compound; the absorbance of such complex compound is proportional to the ammonia nitrogen contents, then determine the absorbance at the wavelength of 420nm.

3 Interference and Elimination

When water sample contains suspended solids, residual chlorine, metal ions such as calcium-magnesium, sulfides and organic substances, then they may generate interference and needs proper treatment, so as to eliminate the influence on the determination.

If there is residual chlorine in the sample, add proper amount of sodium thiosulfate solution to remove; use potassium iodide-starch test paper to test whether the residual chlorine was eliminated completely. When colorization, add proper amount of potassium sodium tartrate solution, which can eliminate the interference from metal ions such as calcium-magnesium. If the sample is thick or colorful, it can be treated with pre-distillation or flocculation methods.

Weigh 20g of hydrochloric acid; dissolve into water; dilute to 1L.

4.12 Bromthymol blue indicator, $\rho=0.5\text{g/L}$.

Weigh 0.05g of bromthymol blue; dissolve into 50ml of water; add 10ml of anhydrous ethanol; then use water to dilute to 100ml.

4.13 Potassium iodide-starch test paper

Weigh 1.5g of soluble starch into a beaker; use small amount of water to make it into paste state; add 200ml of boiling water; stir-mix evenly, and cool down. Add 0.50g of potassium iodide (KI) and 0.50 g of sodium carbonate (Na_2CO_3); use water to dilute to 250ml. After immersion of the filter paper, take it out and get dry; then seal-store in the brown bottle.

4.14 Ammonia nitrogen standard solution

4.14.1 Standard stock solution of ammonia nitrogen, $\rho_{\text{N}}=1000\mu\text{g/ml}$.

Weigh 3.8190g of ammonium chloride (NH_4Cl , guaranteed reagent, dry for 2h at $100\sim 105^\circ\text{C}$); dissolve into water; move to 1000ml flask; dilute to the marked-scale; it can be stored for 1 month at $2\sim 5^\circ\text{C}$.

4.14.2 Ammonia nitrogen standard working solution, $\rho_{\text{N}}=10\mu\text{g/ml}$.

Absorb 5.00ml of ammonia nitrogen standard stock solution (4.14.1) into a 500ml flask; dilute to the marked-scale. It shall be prepared before use.

5 Apparatus and Equipment

5.1 Visible-light spectrophotometer: with 20mm cuvette.

5.2 Ammonia nitrogen distillation device: consists of 500ml Kjeldahl flask, nitrogen ball, straight condenser and catheter; the end of condenser can be directly connected with certain length of dropper, so that the outlet tip is immersed under the absorption liquid surface. 500ml distillation flask can also be used.

6 Samples

6.1 Sample collection and preservation

Collect water sample into the polyethylene or glass bottle; analyze as soon as possible. If it needs preserving, add sulfuric acid so as to ; make the acidify the water sample till $\text{pH}<2$; it can be stored for 7d at $2\sim 5^\circ\text{C}$.

9 Accuracy and Precision

For the standard solution of which the ammonia nitrogen concentration is 1.21mg/L, the repeatability limit is 0.028mg/L, the reproducibility limit is 0.075mg/L, and the recovery rate is 94%~104%.

For the standard solution of which the ammonia nitrogen concentration is 1.47mg/L, the repeatability limit is 0.024mg/L, the reproducibility limit is 0.066mg/L, and the recovery rate is 95%~105%.

10 Quality Assurance & Quality Control

10.1 The absorbance of reagent blank shall not exceed 0.030 (10mm cuvette).

10.2 Preparation of Nessler's reagent

In order to guarantee the good color developing ability of the Nessler's reagent, during the preparing period, it must control the adding amount of HgCl_2 , till the trace HgI_2 precipitate no longer dissolve. For preparing 100ml of Nessler's reagent, the ratio between HgCl_2 and KI is about 2.3:5. During preparing, in order to accelerate the reaction speed and save the preparation time, it can be carried out under low-temperature heating, so as to prevent that HgI_2 red precipitate appears in advance.

10.3 Preparation of potassium sodium tartrate

When, in potassium sodium tartrate, the content of ammonium salt is high, ammonia can't be removed thoroughly only through heating, boiling, or adding Nessler's reagent for precipitation. In this case, add small amount of sodium hydroxide solution; boil-evaporate off 20%~30% of solution volume; after cooling, use ammonia-free water to dilute to the original volume.

10.4 Flocculation precipitation

Filter paper contains certain amount of soluble ammonium salt; in quantitative filter paper, the content is higher than that of qualitative filter paper; qualitative filter paper is recommend to be used for filtering; before filtering, use ammonia-free water, in small amount, to spray several times (generally 100ml). Thus, it can reduce or avoid the measurement error caused by the filter paper.

10.5 Pre-distillation of water sample

During the distillation period, some organic substances may be distilled, together with ammonia, which may interfere the determination; thereof some substance (such as formaldehyde) can be removed through boiling under the acidic condition

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Contact: Wayne Zheng, Sales@ChineseStandard.net

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