Supelco®
Analytical Products

Application Note

Ammonium in effluents with high COD value

Photometric determination using the indophenol blue method

Introduction

Ammonium is a key parameter in water testing, as it can have adverse effects on marine ecosystems if it is at high levels. One major issue is eutrophication, excessive plant and algal growth.[1] As a result, determining ammonium levels in effluents is a critical to maintaining aquatic environments. In this application note, ammonium is quantified in effluents with high chemical oxygen demand (COD) using the Spectroquant® photometric system with ammonium test kits.

As the decomposition with the Crack-Sets 10 or 10C (Potassium peroxodisulfate) fails with strongly organic loaded water (criteria: COD content >300mg/l), a pre-decomposition to destroy the organic matrix is advisable, as described below.

Experimental

Method

Ammonium nitrogen (NH_4-N) occurs partly in the form of ammonium ions and partly as ammonia. A pH-dependent equilibrium exists between the two forms.

In strongly alkaline solution ammonium nitrogen is present almost entirely as ammonia, which reacts with hypochlorite ions to form monochloramine. This in turn reacts with a substituted phenol to form a blue indophenol derivative that is determined photometrically. Due to the intrinsic yellow coloration of the reagent blank, the measurement solution is yellow-green to green in color.

The method is analogous to EPA 350.1, APHA 4500-NH_3 F, ISO 7150-1, and DIN 38406-5.

Reagents and Instruments

Cat. No.	Product Description
Test Kits	
1.14739	Spectroquant® Ammonium Cell Test method: photometric 0.010 - 2.000 mg/l NH_4-N; 0.01 - 2.58 mg/l NH_4^+ or
1.14558	Spectroquant® Ammonium Cell Test Method: photometric 0.20 - 8.00 mg/l NH_4-N; 0.26 - 10.30 mg/l NH_4^+ or
1.14544*	Spectroquant® Ammonium Cell Test Method: photometric 0.5 - 16.0 mg/l NH_4-N; 0.6 - 20.6 mg/l NH_4^+ or
1.14559	Spectroquant® Ammonium Cell Test Method: photometric 4.0 - 80.0 mg/l NH_4-N; 5.2 - 103.0 mg/l NH_4^+ or
1.14752	Spectroquant® Ammonium Test Method: photometric 0.010 - 3.00 mg/l NH_4-N; 0.013 – 3.86 mg/l NH_4^+
Instruments	
1.73026	Spectroquant® VIS Spectrophotometer Prove 100 plus or
1.73027	Spectroquant® UV/VIS Spectrophotometer Prove 300 plus or
1.73028	Spectroquant® UV/VIS Spectrophotometer Prove 600 plus or
1.09748	Spectroquant® Photometer NOVA 30 or
1.09751	Spectroquant® Photometer NOVA 60 or
1.09752	Spectroquant® Photometer NOVA 60A or
1.73632	Spectroquant® Colorimeter Move 100
Materials	
1.14946	Rectangular cells 10 mm or
1.14947	Rectangular cells 20 mm or
1.14944	Rectangular cells 50 mm
1.00731	Sulfuric acid 95 -97 % for analysis
1.05590	Sodium hydroxide solution 32 % for analysis
1.07209	Hydrogen peroxide (Perhydrol®) 30 % for analysis
1.16754	Water for analysis

*not compatible with Move 100
Also first generation Prove instruments are compatible and preprogrammed with this method.

MilliporeSigma

Analytical Approach

Sample preparation

Evaporate 100 ml of sample and 5 ml of sulfuric acid 95 - 97 % in a 600-ml glass beaker to about 10 ml. Mix this mixture **cautiously** with 5 ml Perhydrol® 30% and heat. After the sometimes-heavy reaction (gas formation, foaming) has faded, heat until SO_3-formation starts. If the solution turns brown or dark, let it cool down and add again 5 ml Perhydrol® 30 %. Once more heat until beginning SO_3-formation. If the solution stays colorless or light yellow, let cool down, dilute with about 50 ml distilled water and neutralize the solution with about 14 - 15 ml sodium hydroxide solution 32 % to pH 4 - 6 using a pH - meter. After cooling transfer the solution into a 100-ml volumetric flask and fill up to volume with distilled water and mix.

Ammonium will be fixed as ammonium sulfate in the solution and after the oxidation of the digestion can be measured with the above-mentioned test kits.

Analysis

Determine with the above-mentioned test kits.

Calculation

Ammonium content in mg/l NH_4-N = analysis value in mg/l NH_4-N.

References

- Camargo JA, Alonso A. Ecological and toxicological effects of inorganic nitrogen pollution in aquatic ecosystems: A global assessment. Environ Int. Aug 2006;32(6):831-49. doi:10.1016/j.envint.2006.05.002.

MilliporeSigma
400 Summit Drive
Burlington, MA 01803

To place an order or receive technical assistance

In the U.S. and Canada, call toll-free 1-800-645-5476
For other countries across Europe and the world, please visit: **EMDMillipore.com/offices**
For Technical Service, please visit: **EMDMillipore.com/techservice**

EMDMillipore.com

MS_AN8759EN Ver. 1.0
39131
01/2024