



Parameter

Ammonia in Wastewater (0.01 – 1 mg/L)

Sample Type

Wastewater

Introduction

Ammonia nitrogen in wastewater over 0.01 - 1mg NH₃-N/L range is determined by direct measurement with the Orion 9512HPBNWP ammonia electrode on the Orion Star meter. This procedure is adapted from EPA Approved Standard Methods 4500-NH₃ D. Per Standard Methods 4500-NH₃ D.1.b, sample distillation is unnecessary. Check with local regulators when testing for reporting purposes.

References

1. Method 4500-NH₃ D. Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998. APHA, AWWA, & WEF, Washington, D.C.

www.standardmethods.org

2. D1426-98,03 (B). Annual Book of ASTM Standards, Section 11, Water and Environmental Technology, Volume 11.01. ASTM International, West Conshohocken, PA.

www.astm.org

Result Statistics

# Trials	Average	%CV
4	0.220 mg/L	8.5%

Recommended Equipment

Orion Star Benchtop Meter (Orion 1115000 or Orion 1119000); Ammonia electrode (Orion 9512HPBNWP); Stirrer (Orion 096019); Optional: printer (Orion 1010006); Star Navigator Software (Orion 1010007).

Required Solutions

Ammonia Standards, 100 ppm as N (Orion 951207); Alkaline Reagent (Orion 951011) as ISA; Filling Solution (Orion 951209); deionized water (DI).

Solutions Preparation

1. Prepare 1mg/L ammonia standard by pipetting 1 mL of 100 mg/L (ppm) standard into a 100 mL volumetric flask and diluting to the mark with deionized water.
2. Prepare 0.1 mg/L ammonia standard by pipetting 10 mL of 1 mg/L standard into a 100 mL volumetric flask and diluting to the mark with deionized water.
3. Prepare 0.01 mg/L ammonia standard by pipetting 10 mL of 0.1 mg/L standard into a 100 mL volumetric flask and diluting to the mark with deionized water.
4. Measure 50 mL of each of the three standards into a respective beaker. Do not add Alkaline Reagent (ISA) until just prior to analysis.

Meter Setup

Connect the electrode and stirrer to the meter.

Set measurement mode to ISE. Set stir speed to 4 and read type to continuous. In ISE Setup mode, set resolution to 3, units to mg/L, range to low if the meter's software revision is below 2 or to high range with the revision 2 and above; enable a blank correction by setting "nLin" option to AUTO. If all steps were followed correctly, the meter display will show three digits in the top line and "ISE: mg/L" to the right of the top line.

Electrode Performance Check

Check if slope is between 54-60 mV at least daily according to the low level procedure in the electrode manual. Check electrode drift by comparing a 2 and 3 -minutes reading of 1 mg/L standard w/ISA. The difference in readings should be less or equal 0.5mV. See notes on page 2 or troubleshooting section of manual if slope or drift problems.

Electrode Storage, Soaking, and Rinsing

Before testing, condition electrode for 15 min. in 1mg/L ammonia standard with ISA. Between measurements, rinse the electrode by immersing in two beakers of DI water in sequence. See electrode manual for long term storage. Store electrode overnight in electrode filling solution.

Sample Preservation

Analyze immediately (w/in 15 min.) after collection or acidify to pH <2 with sulfuric acid and store at ≤ 6°C for up to 28 days. Refer to reference and/or EPA 40 CFR Part 136.3 for details.

Sample Preparation

For precise measurements, allow all the standards and the samples to reach the same temperature before analysis. Measure 50 mL sample into a beaker. Just prior to analysis, add 0.5 mL of ISA to 50 mL sample. If the sample pH is not between 11 – 13, add ISA drop wise until necessary pH is achieved. Analyze immediately to avoid loss of ammonia from the alkaline solution.

Calibration

Perform a three point calibration using 0.01mg/L, 0.1mg/L, and 1 mg/L ammonia standards. If takes longer than 5-6 min. to stabilize at 0.01 mg/L, abort the calibration, switch setup range to high, and restart the calibration. The electrode slope will be displayed and should be above 54 mV/decade. Verify the calibration by analyzing a fresh portion of a mid-range standard. If reading is not acceptable, see troubleshooting section of electrode manual.

Analysis

Rinse electrode and stirrer by immersing in two beakers of DI water in sequence and place in the prepared sample. Turn on the stirrer. The "ISE:mg/L" icon will flash until the reading is stable. Press the MEASURE key to print and log the result. Press STIRRER key to turn off stirrer.

Quality Control (See on page 2)



Ammonia in Wastewater	mg/L as Nitrogen
Sample 1	0.248
Sample 2	0.211
Sample 3	0.211
Sample 4	0.210
Mean	0.220
Standard Deviation	0.019
%CV	8.5%

Quality Control (QC)

Recommended QC procedures include: calibration and calibration verification, initial demonstration of laboratory capability and method detection limit determination, laboratory control samples (LCS), method blanks, matrix spikes (MS), sample duplicates, and independent reference materials. See references above for details.

Note:

1). With Auto-blank correction activated, slope on the meter's display can be higher than the expected range of -54 and -60mV/decade due to auto-blank calculation.

2). If sample concentration is outside the calibrated concentration range, dilute the sample into range and rerun.

Alternately, analyze high level samples against a new high level calibration (2-pt). See Applications Log # 17.

3). If the electrode does not give reproducible results or demonstrates a slow response at concentrations greater or equal 0.1mg/L, do the following: be sure that there are no bubbles on the membrane surface. If the electrode performance is not improved, change membrane and filling solution, condition in 1mg/L (w/ISA) standard for 15 minutes and re-calibrate the electrode.