

Ion Selective Electrode Determination of Ammonia Nitrogen in Passaic River Waste Water in New Jersey Essex County Area

Yusuf Yildiz^{1*}, Recep Karadag², Mohammad Cheema³, Mohammad Sayedahmed⁴

¹Analytical Chemistry Research Department, UniChem Analysis Laboratories, Garfield, NJ, USA

²Faculty of Fine Art, Florya Campus, Istanbul Aydin University, Istanbul, Turkey

³Rutgers University, Public Health New Brunswick, NJ, USA

⁴New Jersey Institute of Technology, Chemical Engineering, Newark, NJ, USA

Email: *sayatoglu@yahoo.com

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Abstract

The percent ammonia nitrogen was determined in Passaic River waste water using Ion-Selective Electrode EPA Method 350.3. The intelligent ammonia sensor integrates ammonia electrode, pH electrode and Ammonia Ion electrode together to realize the in situ detection of ammonia. The test results have shown that the sensor is easy operation, low cost and no pollution. The ammonia is determined potentiometrically using an ammonia ion selective electrode and a pH/mV meter, having an expanded millivolt scale. The ammonia selective electrode uses a hydrophobic gas-permeable membrane to separate the sample solution from an electrode internal solution of ammonium chloride. Dissolved ammonia is converted to NH₃ gas by raising the pH to above 11.0 with a strong base. NH₃ gas diffuses the membrane and changes the internal solution pH that is sensed by the electrode. In single laboratory test results have been found 1.001 NH₃-/L and 0.897 mg NH₃-N/L, recoveries were 77.3% and 83.1%, respectively.

Keywords

Ammonia-N, Passaic River Waste Water, Ion Selective Electrode

1. Introduction

The Passaic River is a river, approximately 80 mi (129 km) long [1], in northern New Jersey in the United States. The river in its upper course flows in a highly circuitous route, meandering through the swamp lowlands between the ridge hills of rural and suburban northern New Jersey, called the Great Swamp, drain-

ing much of the northern portion of the state through its tributaries. In its lower portion, it flows through the most urbanized and industrialized areas of the state, including along downtown Newark. The lower river suffered from severe pollution and industrial abandonment in the 20th century. In April 2014, the U.S. Environmental Protection Agency (EPA) announced a \$1.7 billion plan to remove 4.3 million cubic yards ($3.3 \times 10^6 \text{ m}^3$) of toxic mud from the bottom of lower eight miles (13 km) of the river. It is considered one of the most polluted stretches of water in the nation and the project one of the largest clean-ups ever undertaken [2].

The river flows northeast into the city of Paterson, where it drops over the Great Falls of the Passaic. On the north end of Paterson, it turns abruptly south, flowing between Paterson and Clifton on the west and Hawthorne, Fair Lawn, Elmwood Park, Garfield on the east, next through the city of Clifton. At Elmwood Park it begins to form Dundee Lake, created by the Dundee Dam built in 1845. The river becomes navigable two and a half miles downstream of the Dundee Dam at the Eighth Street/Locust Ave Bridge in Wallington where the dredged Wallington Reach channel begins [3]. Proceeding beyond the Wallington Reach, the river remains navigable via a series of maintained channels to its final destination, Newark Bay. It passes Passaic, Clifton again, then Nutley and Belleville on the west; it flows past Rutherford, Lyndhurst and North Arlington to the east.

In its lowest reaches, it flows along the northeast portion of the city of Newark on the west, passing Kearny, East Newark, and Harrison, New Jersey on the eastern bank. Near downtown Newark it makes an abrupt easterly bend, then south around Ironbound, joining the Hackensack River at the northern end of Newark Bay, a back bay of New York Harbor.

Pollution

The Lower Passaic River Watershed suffered severe pollution during the 19th and 20th centuries because of industrial waste discharges such as dioxin, mercury, PCBs, Ammonia, and many other contaminants to the river's sediments and improper waste disposal practices on adjacent land. Although the health of the river has improved due to implementation of the 1972 Clean Water Act and other environmental legislation, and the decline of industry along the river, it still suffers from substantial degradation of water quality. The sediment at the mouth of the river near Newark Bay remains contaminated by pollutants such as dioxin. The dioxin was generated principally by the Diamond Shamrock Chemical Plant in Newark, as a waste product resulting from the production of the Agent Orange defoliation chemical used during the Vietnam War. The cleanup of the dioxin contamination on the bottom of the river is the subject of a major environmental lawsuit regarding the responsibility for the cleanup. In 2008, the EPA reached a settlement with Occidental Chemical Corporation and Tierra Solutions Inc. to clean a portion of the polluted river. A New Jersey Superior Court judge, ruling in July and September 2011, stated that Occidental and

Maxus Exergy Corporation (a subsidiary of YPF) are liable for remediation in other portions of the river [4]. In 2013, several corporate defendants agreed to pay the State of New Jersey \$130 million for ecological damages related to Passaic River pollution. However, it is unclear as to whether the state will actually use this money for clean-up efforts [5].

The New Jersey Department of Environmental Protection (NJDEP) issued notices in 2009 banning commercial fishing and advising the general public that fish caught in the tidal Passaic River (from Dundee Dam to the mouth at Newark Bay) should not be eaten [6]. The fish consumption advisories remain in effect as of 2020 [7].

In April 2014 EPA announced a \$1.7 billion plan to remove 4.3 million cubic yards ($3.3 \times 10^6 \text{ m}^3$) of toxic mud from the bottom of lower eight miles (13 km) of the river. It is considered one of the most polluted stretches of water in the nation and one of the biggest clean-ups project ever undertaken [2].

2. Materials and Method

2.1. Apparatus

Electrometer (pH meter); A pH meter with expanded millivolt scale capable of 0.1 mV resolution between -700 and $+700$ mV such as Hanna Instruments Model 9017.

Ammonia selective electrode, Model NH-35-0002 (Analytical Sensors, Inc. Sugar land TX.) Equipped with a membrane sleeve (part MD-1004-4).

Magnetic stirrer, thermally insulated.

Fluoropolymer coated magnetic bar.

Enviro MIDI-Dist TM (Vineland, NJ) distillation apparatus with the appropriate glassware for distillation [8].

2.2. Reagents

All solutions and standards have been prepared at room temperature and kept in refrigerator at 4°C .

Ammonia Free water (Lab reagent grade water prepared by a DI water system).

Sodium hydroxide, ACS Reagent Grade – 10N. Prepared by dissolving 400 gm NaOH in 800 ml water while stirring. Cool and dilute to 1000 mL with water from ammonia free water.

Stock ammonium chloride solution. Prepared by dissolving 3.819 grams of anhydrous NH_4Cl (ACS Reagent Grade) dried at 100°C for at least one hour in 200 mL while stirring, and dilute to 1000 mL in a volumetric flask.

$$1.0 \text{ mL} = 1.0 \text{ mgN} = 1.22 \text{ mg NH}_3$$

Standard ammonium chloride solutions. Prepare a series of standard solutions covering the concentrations of 50, 25, 10, 5, 1, 0.5 and 0.1 mg $\text{NH}_3\text{-N/Liter}$ by making dilutions of stock NH_4Cl solution from above with water as shown below:

First make a working standard by diluting the stock solution by a factor of 10

Table 1. Dilution of working standard solution.

Working Std. Volume (mL)	Dilute to (mL)	NH ₃ -N Concentration (mg/L)
0.0	100	0.0
0.1	100	0.1
0.5	100	0.5
1.0	100	1.0
5.0	100	5.0
10.0	100	10.0
25.0	100	25.0
50.0	100	50.0

(10 mL in 100 mL).

Now dilute the working std. solution as per **Table 1**.

NaOH/EDTA—10N solution for removing heavy metals that form ammonia complexes. Dissolve 400 gm sodium hydroxide (CAS-1310-73-2) Reagent grade in 800 mL water. Add 45.2 gm Na₄EDTA = 4H₂O (ACS Reagent Grade) and stir to dissolve. Cool and dilute to a 1000 mL in a volumetric flask. Keep all reagents on a dark cool place.

Electrode filling solution supplied by Analytical Sensors Inc. This solution is a precalibrated ammonium chloride solution.

Sodium tetraborate—0.025 M.

Prepare by dissolving 9.5 grams of Na₂B₄O₇·10H₂O, ACS Reagent Grade in 200 mL water and dilute to 1000 ml with ammonia free DI water

Sodium thiosulfate solution (Dechlorinating reagent):

Dissolve 3.5 gm Na₂S₂O₃·5H₂O, ACS Reagent Grade in 1000 mL ammonia free DI water. Add 0.4 mL of chloroform as a preservative.

Note: 1.0 mL of this solution will remove 1 mg/L of residual chlorine in 500 mL sample.

Borate buffer solution:

Add 88 mL 0.1N NaOH solution to 500 mL 0.025 M Na₂B₄O₇ solution and dilute to 1 L with ammonia free DI water

Sulfuric acid—0.04N

Dilute 1.0 ml conc. H₂SO₄ to 1000 ml with ammonia free DI water.

Sodium hydroxide, ACS Reagent Grade—6N:

Dissolve 240 grams NaOH in 800 ml water while stirring. Cool and dilute to 1000 mL with ammonia free DI water.

3. Procedure

Sample Collection:

The sampling has been followed NJDEP guidelines for collection, preservation and transport of water samples. The water samples have been collected from two foot deep flowing water level, and sediment as well in the Passaic River. The 10

laboratory cleaned sample plastic bottle have been used for sampling between 1 mile, from 10 spots of both sides of the river, and then mixed them up to make it 1 L. Sample was preserved with 2 mL of conc. H_2SO_4 per liter and stored at 4°C [9] [10]. The time from sample collection to initiation of analysis may not exceed 28 days. Standard operating Procedure for determination of Ammonia Nitrogen in drinking, surface, and domestic, Industrial waters is based on Method SM-4500-NH₃ B, D [11].

3.1. Scope and Application

This method is applicable to the measurement of ammonia nitrogen in drinking, surface and saline waters and domestic and industrial wastes and wastewater.

This method covers the range from 0.1 to 50 mg NH₃-N per liter.

The application of this method for all samples always requires a preliminary distillation step.

3.2. Interferences

Amines are a positive interference

Mercury and silver interfere by complexing with ammonia. When their presence is suspected add NaOH/EDTA solution.

Residual chlorine must be removed prior to distillation by treatment with sodium thiosulfate in water.

3.3. Distillation Step

Add 50 mL DI water and 2.0 mL borate buffer, adjust pH to 9.5 with 6N NaOH solution, and add to a distillation flask. Add a few glass beads or boiling chips and use this mixture to steam out the distillation apparatus until distillate shows no traces of ammonia [12].

Neutralize sample pH to 7 before distillation. Then, to 50 mL of sample add 2.5 ml borate buffer then add 6 N NaOH dropwise with mixing and adjust to pH 9.5 using a pH meter and record pH on worksheet.

Transfer the sample to the reaction tube, followed by a pinch of boiling chips.

To the receiver tube add 5 mL of 0.04 N H_2SO_4 . The tip of the long stem must be below the H_2SO_4 level.

Assemble the required number of set-ups in the distillation heating block and turn the tap water to cool the cold finger after ensuring all tubing connections are tight. Check for any leak before starting.

Set the block temperature to 190°C and turn heat in.

Collect a minimum of 30 mL of distillate, preferably 40 mL.

Turn the heat off.

Unscrew the cap holding the long stem in the receiver tube and remove from all distillation positions to prevent any suck back of the distilled sample. When the long stem is disconnected gently let it rest at the bottom of the receiver tube.

Dilute the distillate collected in the receiver tube to 50 mL graduation mark

with ammonia free water.

Place a collection tray of individual beakers located directly below where the receiver tubes were removed to catch any further distillate. Distillation will continue because the heater block is still hot.

After each use clean exterior with a damp sponge.

When the heater temperature falls below 100°C, the next run can be started.

3.4. Once Distillation has been Completed Start This Step

Before running standards, check the electrometer calibration as instructed in the instrument manual. If the electrode is functioning properly a tenfold change of $\text{NH}_3\text{-N}$ concentration produces a potential change of about 59 mV.

Place 50 mL of each standard solution in 150 mL beaker. Immerse pH electrode and ammonia electrode in standard of lowest concentration and mix with a magnetic stirrer at low speed to minimize loss of ammonia from the solution. Record pH on worksheet.

Note: Possible loss of ammonia from the solution may cause cross contamination of other standards or sample(s). Handle the analysis in a way to minimize such possible cross contamination.

Maintain the same stirring rate and a temperature of about 25°C throughout the calibration and testing procedures.

Add a sufficient volume of 10N NaOH solution (1 mL is usually sufficient) to raise the pH above 11.0 keeping the electrode in solution until a stable millivolt reading is obtained. Record the amount of NaOH added on the worksheet.

Note: 1) Do not add the NaOH solution before immersing the electrode.

2) If the presence of ammonia complexing metals (Ag or Hg) is suspected add instead 1 ml of NaOH/EDTA, reagent 2.2.4. above.

Repeat the above procedure with all the standards in an increasing order of concentration. Note the volume added.

3.5. Preparation of a Standard Curve

Using the appropriate excel program obtain a plot of ammonia-N concentration in mg/L (on the log axis) vs. potential ion millivolts on the linear axis.

3.6. Measurement of Samples

Dilute if necessary prior to measurement of potential to bring the $\text{NH}_3\text{-N}$ concentration to within the calibration curve range. Follow the procedure 3.4. and 3.5. for 50 mL of sample in 150 mL beakers. Record the stabilized potential of each unknown sample and convert the potential reading to the ammonia concentration using the standard curve regression equation: (via the excel program).

Note: Blank subtraction or correction is NOT permitted in this procedure.

Note: The ammonia selective electrode responds slowly below 1mg $\text{NH}_3\text{-N/L}$; hence use longer time of electrode immersion (2 to 3 min.) to obtain stable readings.

4. Experiments

Sample measurements: Follow the procedure record the stabilized potential of each unknown sample, blank, spiked and spiked sample, read the ammonia level directly in mg NH₃-N/L using Ion Selective Ammonia Electrode (ISE) [13].

NH₃ Selective Electrode Measurements

Most critical for reproducible measurements are good electrical connections between electrode and meter. Hanna uses adapter, picks up noise (± 3 mV when touch benchtop, ± 50 mV when touch ref connection, poor reproducibility day to day). Very good results (noise-free and reproducible) are obtained with JENCO Model-671p meter (battery operated), which provides direct connection of electrode to meter.

Also critical is the membrane. Follow the flow chart carefully. The membrane can be restored to good operating conditions by soaking overnight in DI water (fill with water, and put in beaker containing water).

Preparation: Prepare standards (50, 25, 10, 5 ppm directly from 1 mg/mL; 1, 0.5, 0.1 from 10 ppm). Prepare thymolphthalein (transition 8.3 to 10.5) by dissolving 0.04 g in 50 mL ethanol and adding 50 mL H₂O. Prepare 50% KOH by weighing 250 g KOH pellets (Baker Analyzed Reagent) and adding 250 mL (or 250 g) of H₂O. Mix in water bath in hood. When cool, transfer to plastic bottle. Note: Samples and standards must be adjusted to \geq pH 11 for accurate determination of NH₃ by electrode [14] [15] [16].

Other supplies: Stir plate, small (micro, 10mm length) stir bar, 50 mL beaker, paper towel torn into 8 parts. Holder for electrode. Pipets and volumetric flasks as needed for standards.

Protocol: Prepare electrode as directed. Electrode in DI water should have reading of 175 mV. Put 40 mL (use graduated cylinder) of lowest concentration standard into dry beaker with stir bar, begin stirring (mark dial, turn to same place every time. Approximately 1/2 turn from off position, Corning stir/hotplate.) Lower electrode till it is, "2 - 3 mm" above stir bar. Position should be reproducible when use electrode holder. Add 10 drops of KOH using disposable plastic transfer pipet. After stable reading is achieved (see below), stop stir bar, raise electrode (DON'T MOVE HOLDER) and clamp with large wooden spring clamp. Empty beaker, rinse electrode and beaker with DI water, blot dry with section of paper towel. Continue to next concentration, no need to rinse graduated cylinder as follow increasing trend in standard concentration. Time for stable reading: 0.1 ppm, 5 - 10 min; 0.5 ppm, 2 - 5 min; 1 ppm, 1 - 2 min; all others, a minute or less.

Rinse beaker, electrode, and graduated cylinder thoroughly with DI water. Put DI water in beaker, stir; electrode reading should approach 175 mV.

Plot E vs. \log_{10} [NH₃]/ppm and obtain three slopes and intercepts: one for lowest three concentrations, one for highest three concentrations, and one for all the concentrations. Keep a running tabulation for QC purposes (Table 2).

Additional Notes:

Table 2. Ammonia-N calibration curve data.

NH ₃ -N [mg/L]	Log [NH ₃ -N]	E/mV	R ²	Calc C	Del E
0.1	-1	110.1		0.09	
0.5	-0.3	64.7	1	0.55	
1.0	0	48.5	0.9983	1.04	61.6
5.0	0.7	8.5	0.99833	5.08	56.2
10.0	1	-9.2	0.9989	10.25	57.7
25.0	1.4	-31.1	0.99908	24.43	
50.0	1.7	-48	0.99923	47.76	56.5
m	-58.06	49.49	b		
s_m	0.72	0.74	s_b		
R ²	0.9992	1.72	s_y		

Note: Do not over-tighten outer electrode. It is spring loaded; once connector is threaded, there is no improvement in reading by tightening it. Over-tightening may press inner electrode against membrane causing damage.

Note: Outer body mass is 6.0 g when empty; add 3.5 mL IFS by mass. At end, check membrane integrity by noting mass of outer body + IFS, it should be 9.5 g.

Note: Replace IFS to bottle after measurements. IFS is NH₄Cl solution of approximately 1000 ppm concentration.

Note: See attached notes regarding NH₃ electrode preparation and troubleshooting, as well as preparation of standard solutions.

4.1. Quality Control Checks

4.1.1. Precision Check

Sample: 1.001 mg [NH₃-N]/L, Duplicate result: 0.897 mg [NH₃-N]/L

Average: 0.949 [NH₃-N] mg/L

%RPD calculation:

$$\%RPD = \frac{\text{Sample Result} - \text{Duplicate Result}}{\text{Average}} \times 100 = 10.96\%$$

4.1.2. Accuracy Check

$$\% \text{ Recovery} = \frac{\text{Spiked Sample Result} - \text{Sample Result}}{\text{Actual Known Concentration}} \times 100$$

4.1.3. Calculations

$$\text{mg NH}_3\text{-N/Liter} = A \times B (50.0 + D)/(50.0 + C)$$

where:

A—Dilution factor (as applicable).

B—Concentration of NH₃-N/L in mg/L units as obtained from calibration curve.

C—Volume of 10N NaOH (or 10N NaOH/EDTA, where applicable) added to calibration standards, in mL.

D—Volume of 10N NaOH (or 10N NaOH/EDTA, where applicable) added to sample, in mL.

50—Sample volume in mL.

5. Results and Discussion

Dissolved ammonia-Nitrogen ($\text{NH}_3\text{-N}$) were measured using an ammonia selective electrode, which a pH meter with expanded millivolt scale capable of 0.1 mV, Hanna Instrument Model 9017. Ammonia concentrations were determined by the ammonia selective electrode method [Table 3 and Table 4]. Ammonia-N

Table 3. Samples measurements by ammonia ion selective electrode.

WHAT	DF	E/mV	Log [$\text{NH}_3\text{-N}$]	[$\text{NH}_3\text{-N}$] mg/L
Blank	1	196.9	-2.6	0.003
0.1 mg/L ind	1	103.3	-0.95	0.112
Sample	1	49.3	0	1.001
Sample Dup	1	52.0	-0.05	0.897
Spike	1	35.2	0.25	1.774
Spike Dup	1	34.4	0.26	1.832
Ave	0.95			
Precision	10.96			
Spike amount	0.1			
Recov 1	0.773		0.1 mL \times 1000 mg/L is	
%1	77.3		0.1 mg; in 100 mL	
Recov 2	0.831			
%2	83.1			

Table 4. Ammonia-N Sample Measurements by Ammonia Ion selective Electrode

Test #	Lab ID	Distilled	pH Adjustment (9.50)	Volume of sample distilled (mL)	Final Volume of Distillate (mL)	Distillate pH (2.00)	Volume of NaOH added 1.00 mL	Reading (mV)
1	Blank	No	9.50	50	50	2.0	1.00	196.9
2	Sample	Yes						49.3
3	Sample dup	Yes						52.0
4	0.1 mg/L check std	Yes						103.3
5	Sample Spiked	Yes						35.2
6	Sample Spiked Dup	Yes						34.4
7	10 mg/L Check Std.	Yes						-9.2

has been found 1.001 mg NH₃-N/L and duplicate 0.897 mg NH₃-N/L respectively. Percent spiked and spiked recovery result has been found 77.3% and 83.1% respectively (Table 5). The average result meets the Environmental Protection Agency (EPA) specifications.

6. Quality Assurance/Quality Control

Run a method blank with each set of samples.

Determine precision and accuracy for every 20 samples or once a month if less than 20 samples are analyzed. Precision between the sample and duplicate must be less than 10% and the accuracy for the spike recoveries must be between 80% - 120%.

To determine accuracy run a method spike and spike duplicate. Perform the spiking every 20 samples or once a month if less than 20 samples are analyzed. Add enough spiking solution so that the added concentration (or absolute amount) is within the calibration curve.

A calibration curve must be analyzed every three months with a correlation coefficient of greater than or equal to 0.995 (Figure 1).

A second source QC check must be analyzed every 20 samples or once a month if less than 20 samples are analyzed in that month. Recovery must be within 10% of the true value.

Each day samples are analyzed a check standard from the same source as the curve must be analyzed. Recovery must be within 10% of the true value.

Table 5. Calculation for MS/MSD.

Matrix Spike Recovery	Matrix Spike Recovery Duplicate	Average Spike Recovery
%R _{MS} = 77.3%	%R _{MSD} = 83.1%	%R = 80.2%

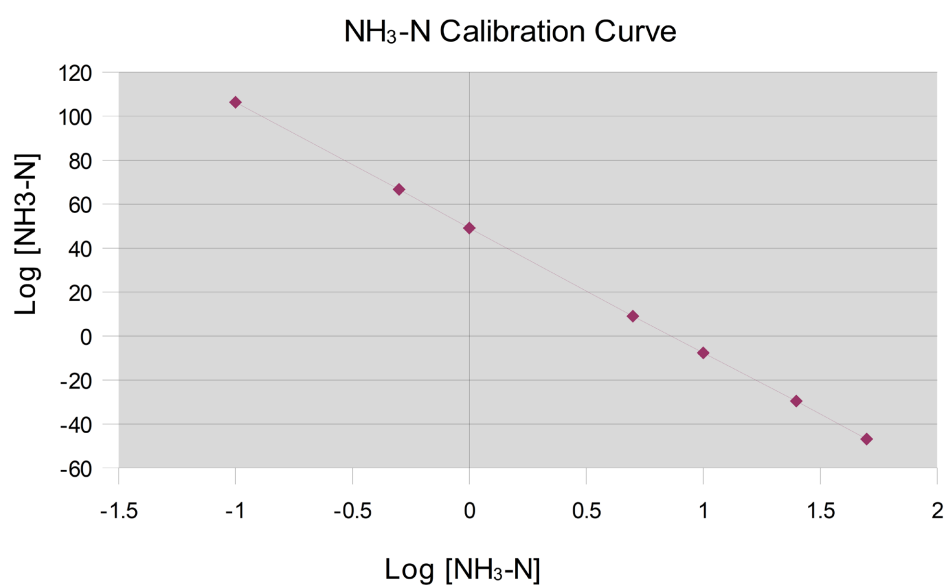


Figure 1. Calibration curve, linear plot of [NH₃-N] concentration.

7. Safety and Waste Disposal

It is desirable to conduct the analysis of unknown samples in a hood to avoid the possibility of generating high concentration of ammonia vapors and to minimize cross contamination of samples.

Solution containing ammonia-N in concentrations higher than 40 mg/L must be treated for ammonia destruction prior to discharge into the sewer line.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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