

Spectroscopic Determination of Percent (w/w) Ammonium Sulfate in Hydroxylamine Sulfate using Ammonia Selective Electrode

BY

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Abstract

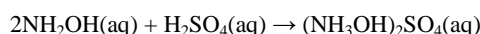
The determine of percent ammonium sulfate were determined in hydroxylamine sulfate using Ammonia Ion-Selective Electrode. The intelligent ammonia sensor integrates ammonia electrode, pH electrode, and Ammonia Ion electrode together to realize the in situ detection of ammonia. The test results have shown 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 0.407 NH₃-N/L and 0.405 NH₃-N/L, recoveries were 96.67% and 96.00%, respectively. The test results meet the FDA specifications.

Keywords: Hydroxylamine sulfate, ammonium sulfate, ammonia selective electrode

1. Introduction

Ammonium sulfate (NH₄)₂SO₄ is an inorganic sulfate salt obtained by reaction of sulfuric acid with two equivalents of ammonia. A high-melting (decomposes above 280°C) white solid which is very soluble in water (70.6 g/100 g water at 0°C; 103.8 g/100 g water at 100°C), it is widely used as a fertilizer for alkaline soils. It has a role as a fertilizer. It is an ammonium salt and an inorganic sulfate salt. Ammonium sulfate is a white odorless solid. Sinks and dissolves in water [1].

Hydroxylamine (NH₂OH) plays an important role in the chemical industry. Hydroxylammonium salts constituting stable form of hydroxylamine are used in many branches of the chemical industry [2]. Hydroxyl ammonium sulfate can be obtained by the acid-base reaction of hydroxylamine with sulfuric acid:



Hydroxylammonium sulfate is used in organic synthesis to convert aldehydes and ketones to oximes, carboxylic acids and their derivatives (e.g. esters) to hydroxamic acids, isocyanates to N-hydroxyureas and nitriles to amidoximes. Hydroxylammonium sulfate is also used to generate

hydroxylamine-O-sulfonic acid from oleum or chlorosulfuric acid.

Hydroxylammonium sulfate or hydroxylamine sulfate (NH₂OH)₂. H₂SO₄) is used in the production of anti-skinning agents, pharmaceuticals, rubber, textiles, organic synthesis, antioxidant in fatty acids, and soaps. plastics and detergents. (NH₂OH)₂SO₄ is a starting material for some insecticides, herbicides, and growth regulators [3]. Hydroxylamine structural formula is shown below.

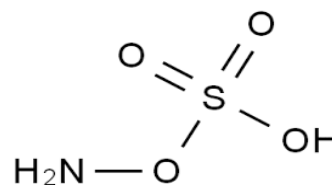
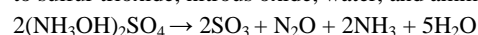


Figure1. Hydroxylamine sulfate (HONH₃)₂SO₄

At 120 °C, hydroxyl ammonium sulfate begins to decompose to sulfur trioxide, nitrous oxide, water, and ammonia:



The reaction is exothermic above 138 °C and is most exothermic at 177 °C. [4] Metals (especially copper, its alloys, and its salts) catalyze the decomposition of hydroxyl ammonium sulfate. The instability of this compound is mainly

due to the hydroxylammonium ion's weak nitrogen-to-oxygen single bond.

2. Materials and method

2.1. Sample Handling and Preservation

- 2.1.1. All samples for Ammonia-N analysis must be preserved with H₂SO₄. Check samples immediately upon arrival in the laboratory for pH. Lower the pH to less than 2 with concentrated H₂SO₄. Refrigerate at 4°C until analysis time.
- 2.1.2. All samples must also be checked for residual chlorine and treated if necessary at time of collection. If residual chlorine is found, 1 mL dechlorinating reagent to remove 1 mg/L residual chlorine in 500 mL sample. The dechlorinating agent is prepared by dissolving 3.5 g sodium thiosulfate (Na₂S₂O₃ • 5H₂O) in water and dilute to 1 L. This should be prepared fresh on a weekly basis.
- 2.1.3. Holding time is 28 days.
- 2.1.4. Minimum sample volume required is 250 mL. Brown glass jar or plastic containers are acceptable.

2.2. Apparatus

- 2.2.1. Electrometer (pH meter): A pH meter with an expanded millivolt scale capable of 0.1mV resolution between -700 and + 700 mV such as Hanna Instruments Model 9017.
- 2.2.2. Ammonia selective electrode, Model NH-35-0002 (Analytical Sensors, Inc. Sugar land TX.) equipped with a membrane sleeve (part MD-1004-4).
- 2.2.3. Magnetic stirrer, thermally insulated.
- 2.2.4. Fluoropolymer-coated magnetic bar.
- 2.2.5. Enviro MIDI-Dist. TM (Vineland, NJ) distillation apparatus with the appropriate glassware for distillation [5].

2.3. Reagents

- 2.3.1. Ammonia Free water (Lab reagent grade water prepared by a DI water system).
- 2.3.2. Sodium hydroxide, ACS Reagent Grade – 10N. Prepared by dissolving 400 g NaOH in 800 ml water while stirring. Cool and dilute to 1000 mL with ammonia-free water.
- 2.3.3. Stock ammonium chloride solution. Prepared by dissolving 3.819 grams of anhydrous NH₄Cl (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 mL=1.0 mg N=1.22 mg NH₃
- 2.3.4. 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 NH₃-N/Liter by making dilutions of stock NH₄Cl solution from 2.3.1. above with water as shown below:

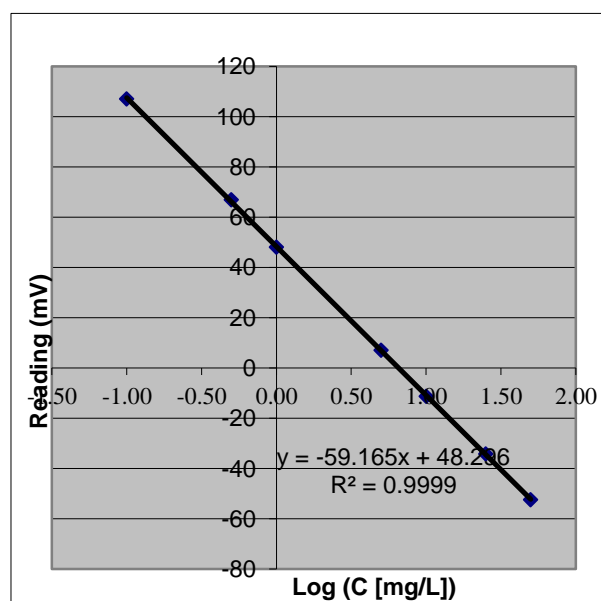
First, make a working standard by diluting the stock solution (2.3.1.) by a factor of 10 (10mL in 100mL).

- 2.3.5. Now dilute the working std. solution as per the table below:

Table1: Dilution of Working Standard Solution

Working std. Volume (mL)	Dilute to: (mL)	NH3-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

Figure2: Calibration curve, linear plot of [NH₃-N] concentration



- 2.3.6. A second source check standard of 0.1 ppm will be prepared using a different lot of anhydrous NH₄Cl (ACS Reagent Grade) as described in 2.3.1. above.
- 2.3.7. 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 45.2g 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.

- 2.3.8. Electrode filling solution supplied by Analytical Sensors Inc. This solution is a precalibrated ammonium chloride solution.
- 2.3.9. Sodium tetraborate – 0.025M. Prepare by dissolving 9.5 grams of $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10 \text{H}_2\text{O}$, ACS Reagent Grade in 200 mL water and dilute to 1000 ml with ammonia-free water.
- 2.3.10. Sodium thiosulfate solution (Dechlorinating reagent): Dissolve 3.5 gm $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$, ACS Reagent Grade in 1000 mL with ammonia-free water. Note: 1.0 mL of this solution will remove 1 mg/L of residual chlorine in 500mL sample.
- 2.3.11. Borate buffer solution: Add 88 mL 0.1N NaOH solution to 500 mL 0.025M $\text{Na}_2\text{B}_4\text{O}_7$ solution (section 2.3.9.) and dilute to 1L with ammonia-free water.
- 2.3.12. Sulfuric acid – 0.04N Dilute 1.0 ml conc. H_2SO_4 to 1000 ml with ammonia-free water.
- 2.3.13. Sodium hydroxide, ACS Reagent Grade – 6N: Dissolve 240 grams NaOH in 800 ml water while stirring. Cool and dilute to 1000mL with ammonia-free water.
- 3.3.4. 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.
- 3.3.5. 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.
- 3.3.6. Set the block temperature to 190°C and turn heat in.
- 3.3.7. Collect a minimum of 30 mL of distillate, preferably 40 mL.
- 3.3.8. Turn the heat off.
- 3.3.9. 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 receive tube.
- 3.3.10. Dilute the distillate collected in the receiver tube to 50 mL graduation mark with ammonia-free water.
- 3.3.11. 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.
- 3.3.12. After each use clean exterior with a damp sponge.
- 3.3.13. When the heater temperature falls below 100°C, the next run can be started.

3. Procedure

Standard operating Procedure for determination of Ammonia Nitrogen in drinking, surface, and domestic, Industrial waters is based on Method SM-4500-NH₃ B, D [6].

3.1. Scope and Application

- 3.1.1. This method is applicable to the measurement of ammonia nitrogen in drinking, surface, and saline waters and domestic and industrial wastes and wastewater.
- 3.1.2. This method covers the range from 0.1 to 50 mg NH₃-N per liter.
- 3.1.3. The application of this method for all samples always requires a preliminary distillation step.

3.2. Interferences

- 3.2.1. Amines are a positive interference
- 3.2.2. Mercury and silver interfere by complexing with ammonia. When their presence is suspected add NaOH/EDTA solution.
- 3.2.3. Residual chlorine must be removed prior to distillation by treatment with sodium thiosulfate in water.

3.3. Distillation step

- 3.3.1. 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.
- 3.3.2. Neutralize sample pH to 7 before distillation. Then, to 50 mL of sample add 2.5 ml borate buffer then add 6N NaOH dropwise with mixing and adjust to pH 9.5 using a pH meter and record pH on the worksheet.
- 3.3.3. Transfer the sample to the reaction tube, followed by a pinch of boiling chips.

3.4. Once distillation has been completed start this step.

- 3.4.1. Before running standards, check the electrometer calibration as instructed in the instrument manual. If the electrode is functioning properly a tenfold change of NH₃-N concentration produces a potential charge of about 59 mV.
- 3.4.2. Place 50 mL of each standard solution in 150 mL beaker. Immerse pH electrode and ammonia electrode in the standard of lowest concentration and mix with a magnetic stirrer at low speed to minimize loss of ammonia from the solution. Record pH on the worksheet. 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.
- 3.4.3. Add a sufficient volume of 10N NaOH solution (1 mL is usually sufficient) to raise the pH above 11.0 keeping the electrode in the solution until a stable millivolt reading is obtained. Record the amount of NaOH added on the worksheet. Do not add the NaOH solution before immersing the electrode. If the presence of ammonia complexing metals (Ag or Hg) is suspected add instead 1 ml of NaOH/EDTA, reagent 2.3.8. above.

- 3.4.4. Repeat the above procedure with all the standards in 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 NH₃-N concentration to within the calibration curve range. Follow the procedure 3.4. and 3.5. for 50mL of sample in 150mL beakers. Record the stabilized potential of each unknown sample a convert the potential reading to the ammonia concentration using the standard curve regression equation: (via the excel program).
- 3.6.1. Blank subtraction or correction is NOT permitted in this procedure.
- 3.6.2. The ammonia selective electrode responds slowly below 1mg NH₃-N/L; hence use longer time of electrode immersion (2 to 3 min.) to obtain stable readings.

4. Experimental

Sample measurements: Follow the [procedure to record procedure-record](#) the stabilized potential of each unknown sample, blank, spiked and spiked sample. ~~Then,~~ read the ammonia level directly in mg NH₃-N/L using Ion Selective Electrode (ISE) [7]. An Orion 95-10 ammonia-selective electrode was used with an Orion 601 A pH and millivolt meter (Orion Research Cambridge, MA).

~~All solution~~ [The entire solution](#) was prepared in distilled deionized water. Working solutions of ammonia were prepared fresh in 10 M sodium hydroxide just prior to each set of experiments.

Three types of experiments are described. In the first and second set, the electrode was equilibrated in 0.1 and 0.003 M ammonia, respectively, between samples; in the set, the electrode was equilibrated between samples in different randomly selected concentrations of ammonia between 0.0001 and 0.1 M. The electrode was rinsed with water and wiped lightly with tissue as it was transferred from the equilibration solution to each sample. All samples were monitored to steady state so that computed values of steady-state potentials could be compared with measured values [8].

4.1. NH₃ Selective Electrode Measurements

Most critical for reproducible measurements are good electrical connections between electrode and meter. Hanna uses an 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.~~ [The membrane is also quite critical.](#) ~~FF~~ Follow the flow chart carefully. The membrane can be restored to good operating conditions by soaking overnight

in DI water (filled with ~~water, and water and~~ put in [a](#) beaker containing water).

Preparation: Prepare standards (50, 25, 10, 5 ppm directly from 1mg/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 [a](#) water bath in [the](#) 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. [9] [10] [11].

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

Protocol: Prepare electrode as directed. [The e](#)Electrode in DI water should have [a](#) 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, [CC](#)orning stir/hotplate.) Lower electrode till it is 2-3mm above stir bar. Position should be reproducible when using [e](#)electrode holder. Add 10 drops of KOH using disposable plastic transfer pipet. After stable reading is achieved (see below), stop stir bar, raise electrode (do not 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 to~~ follow an 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.

~~Next, r~~inse [the](#) beaker, electrode, and graduated cylinder thoroughly with DI water. Put DI water in beaker, stir; electrode reading should approach 175 mV.

Plot E vs. $\log_{10}[\text{NH}_3]/\text{ppm}$ and obtain three slopes and intercepts: one for lowest three concentrations, one for the highest three concentrations, and one for all the concentrations. Keep a running tabulation for QC purposes (Table2).

Additional Notes:

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.

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.

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

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

Sample preparation: Sample 1.00 g was dissolved in water, put to 250 mL VF, QF H₂O. It was distilled in Enviromididist.

It was tested using NH₃ selective electrode. [NH₃] was near the top of the calibration curve; which was unexpected. [Maybe-It is possible that](#) the sample decomposes to yield NH₃. Merck Index-hydroxylamine decomposes in hot water. [Again-p](#)Put 1.00 g sample in water [once again](#), 250 mL VF, QF H₂O. Now measure NH₃ directly without distillation by Hanna meter.

- 4.1. Run a method blank with each set of samples.
- 4.2. 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%.
- 4.3. 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.

- 4.4. A calibration curve must be analyzed every three months with a correlation coefficient greater than or equal to 0.995.
- 4.5. 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.
- 4.6. 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.

Table2: Ammonia-N calibration curve data-1

Standard Conc. (mg/L)		0.10	0.50	1.00	.00	10.00	25.00	50.00
Log Conc.		-1.00	-0.30	0.00	0.70	1.00	1.40	1.70
Reading (mV)		107	66.9	48.1	7.1	-11.2	-34.2	-52.4
m	-59.35	48.49	b					
s-m	0.15	0.16	s-b					
	1.0000	0.37	s-y					
	148695	5.00						
	19989.94	0.67						

A Squared (from instrument): 0.9999
 R Squared (from Excel): 0.9999
 Slope: -59.1651
 Intercept: 48.2961

Figure2: NH₃-N Calibration Curve, linear plot of [NH₃-N] concentration

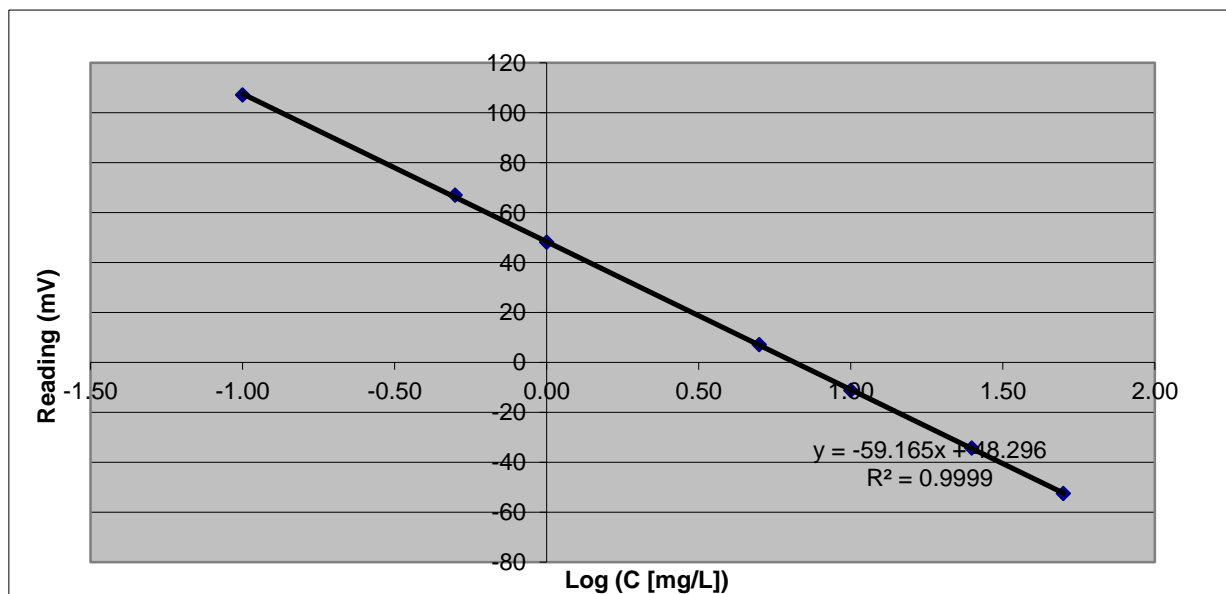


Table3: Samples measurements by Ammonia Ion Selective Electrode

Test	What/Lab ID	Distilled	Dilution Factor (DF)	E/mV	log[NH ₃ -N]	[NH ₃ -N] mg/L
1	Blank/ Ammonia free DI.Water	No	1	188.0	-2.35	0.004
2	Sample	Yes	1	-29.9	1.32	41.861
3	Sample dup	Yes	2.5	-23.5	1.21	40.821
4	10 mg/L Check Std.	yes	1	-11.9	1.02	10.411
5	Sample	No	1	71.7	-0.38	0.407
	sample dup	No	1	71.8	-0.39	0.405
6	Sample spike1	No	1	63.9	-0.26	0.550
7	Sample spike2	No	1	63.8	-0.27	0.549
8	MDL 0.05 mg/L	No	1	125.0	-1.29	0.051

5. Result and Discussion

Dissolved Ammonia-Nitrogen (NH₃-N) ~~were~~ was measured using an ammonia selective electrode, which a pH meter with expanded millivolt scale capable of 0.1 mV and a –Hanna Instrument model 9017. Ammonia concentrations were determined by the ammonia-selective electrode method [Table 3]. Percent (w/w) NH₃-N test results have been found 0.407 NH₃-N/L and 0.405 NH₃-N/L in Hydroxylamine Sulfate. Percent recoveries were 96.67% and 96.00%, respectively. The test results meet the FDA specifications.

CALCULATIONS

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

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.

5.1. QUALITY ASSURANCE/QUALITY CONTROL

Before spike: 1st 0.407 mg NH₃/L
dup 0.405-dropped

$$\text{ave.} = 1/2(0.407 + 0.405) = 0.406$$

$$\% \text{ RPD} = \frac{\text{sample result} - \text{duplicate result}}{\text{Average}} \times 100 \quad \text{RPD} = 0.49\%$$

Actual Known Concentration: 1.15 mL x 1000 mg/L is 0.15 mg, in 100 mL

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

Table4: Calculation for MS/MSD

Matrix Spiked Recovery	Matrix Spiked Duplicate Recovery	Average Spiked Recovery
%R _{MS} = 96.67%	%R _{MSD} = 96.00%	%R= 96.34%

5.2. Quality Control Sample (QCS):

Analyze ~~and~~ externally generated, blind proficiency testing sample at least annually. Obtain this sample from a source external to the laboratory, and compare results to that laboratory’s acceptance results.

5.3. Method detection level (MDL):

Verify MDL at least annually. MDL for this method is usually below 0.1 mg/L.

5.4. Method blank (MB):

Include at least one MB daily or with each batch of 20 or fewer samples tested on the same day, whichever is more frequent.

5.5. QC Check Standards

A second source standard verification that must be performed at least once a month or every 20 samples tested when more than 20 samples are tested in a month. It is to be accurate within 15% of the true value.

Include a Laboratory Fortified Blank (LFB) daily or with each batch of 20 or fewer samples, whichever is more frequent. The LFB must agree within 10% of its true value.

5.6. Duplicates:

Include at least one duplicate for each sample daily or with each batch of 20 or fewer samples. Run either a sample duplicate or an LFMD per batch. RPD must be ≤10%

5.7. Matrix Spike(MS)/ Matrix Spike Duplicate (MSD):

Include at least one LFM/LFMD daily or with each batch of 20 or fewer samples. This must be control charted.

5.8. Calibration:

Calibrate with five calibration standards of analyte(s) of interest. Apply linear to analyze the concentration-instrument response relationship. The appropriate linear correlation coefficient for standard concentration-to-instrument-response must be greater than or equal to 0.995.

5.9. Calibration Verification:

Verify calibration by analyzing a calibration standard and calibration blank after each batch of ten samples and at the end of the run.

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

5.10. Initial Quality Control

Before new analysts run any samples, verify their capability with the method. Run a laboratory-fortified blank or a standard with known or certifiable concentration at least 4 times and compare to the limits listed in the method.

6. Safety and Waste Disposal

- 6.1. 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.
- 6.2. Solution containing Ammonia-N in concentrations higher than 40 mg/L must be treated for ammonia destruction prior to discharge into the sewer line.

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