Askew Scientific Consulting LLC

TIMBERLINE INSTRUMENTS

Timberline Ammonia-001

Determination of Inorganic Ammonia by Continuous Flow Gas Diffusion and Conductivity Cell Analysis

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1. SCOPE and APPLICATION

1.1. Ammonia gas permeation through a hydrophobic membrane is used to determine ammonia concentration in aqueous solutions.

1.2. For the determination of total ammonia from solutions that contain organic nitrogen (Kjeldahl nitrogen) and inorganic ammonia in aqueous and solid samples, a Kjeldahl digestion is required prior to analysis.

1.3. Detection limits and linear ranges for organic and inorganic ammonia will vary with the matrices. Tables B and C in Section 16 provide pooled results for method detection limit (MDL) and minimum level (ML) determined from the inter-laboratory method validation study for 4 inch or 10 inch membranes. However, actual method detection limits and linear working ranges will be dependent on the sample matrix, instrumentation, and selected operating conditions.

1.4. Users of the method data should state the data-quality objectives prior to analysis. Users of the method must document and have on file the required initial demonstration of capability data described in Section 9.2 prior to using the method for analysis.

2. SUMMARY

2.1. An aqueous sample is combined with 1-15% sodium hydroxide to attain a pH above 11 producing ammonia in a non-ionized form in solution.

$$NH_4^+{}_{(aq)} + OH^-{}_{(aq)} \rightarrow NH_{3(g)} + H_2O_{(aq)}$$

2.2. This solution is conveyed to a membrane assembly and the gaseous ammonia in the aqueous sample migrates through the hydrophobic membrane into the borate buffer absorption solution, which is transported to a conductivity cell.

2.3. The measured changes in conductivity are used to quantitate ammonia in the sample using an external calibration.

3. DEFINITIONS

3.1. Ammonia Stock Standard Solution: A concentrated solution containing method analyte prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

3.2. Calibration Blank: A volume of reagent water acidified with the same acid matrix as in the calibration standards. The calibration blank is a zero standard and is used to calibrate the ammonia analyzer.

3.3. Calibration Standard: A solution prepared from the dilution of stock standard solutions. These solutions are used to calibrate the instrument response with respect to analyte concentration.

3.4. Continuing Calibration Verification (CCV): Ammonia standard that has a concentration between the lower calibration standard and upper calibration standard. A CCV will be run at least once per batch.

3.5. Control Charts: Graphical charts that contain the expected value (the central line) and the acceptable range of occurrence. The acceptable range is determined from the control limits and warning limits. Refer to Part 1000 of Standard Methods for the Examination of Water and Wastewater for further explanation and guidance.

3.6. Dynamic Range (DR): The concentration range over which the instrument response to an analyte is second order quadratic. This range is defined by the concentration range between the lowest concentration standard and the highest concentration standard.

3.7. Instrument Detection Limit (IDL): The concentration equivalent to the analyte signal which is equal to replicate measurements of the calibration blank.

3.8. Initial Demonstration of Capability (IDC) also called a Initial Precision and Recovery (IPR): IDC/IPR are run by analysts with no experience with this method before they run any samples to verify their capability with the method or when significant maintenance or modifications are performed on the instrument. A laboratory fortified blank (LFB) is analyzed four times, the mean recovery and standard deviation are calculated and compared to the limits listed in this method for IDC/IPR.

3.9. Instrument Performance Check (IPC) Solution: A solution of method analyte, used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

3.10. Laboratory Fortified Blank (LFB): An aliquot of ammonia free reagent water to which known quantities of ammonia is added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

3.11. Laboratory Fortified Sample Matrix/Duplicate (LFM/LFMD) also called a Matrix Spike/Duplicate (MS/MSD): An aliquot of an environmental sample to which known quantities of ammonia is added in the laboratory. The LFM/LFMD are analyzed exactly like a sample, and there purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM/LFMD corrected for background concentrations.

3.12. Laboratory Reagent Blank (LRB): An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, reagents or apparatus.

3.13. Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. Full requirements are listed in 40 CFR Part 136 Appendix B.

3.14. Minimum Level (ML): Minimum level is determined by multiplying the MDL by 3.18 and rounding the product to the number nearest to 1 or 2 or 5 x 10^n, where n is a positive or negative integer. The minimum level is used to determine the lowest standard concentration that can be used for the instrument.

3.15. Ongoing Demonstration of Capability (ODC) also called Ongoing Precision and Recovery (OPR): ODC/OPR are performed at least once per sample batch to demonstrate proficiency with the method. Reagent water is spiked with known quantities of ammonia. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this method for precision and recovery.

3.16. Sample Batch: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch. A batch cannot span between laboratory work days (24 hrs). New batches must be started each laboratory work day.

3.17. Solid Sample: For the purpose of this method, a sample taken from material classified as soil, sediment or sludge that is digested by the Total Kjeldahl Nitrogen method.

3.18. Total Kjeldahl Nitrogen: Digestion method of water or solid samples by methods currently approved in 40 CFR 136, Table IB.

3.19. Water Sample: For the purpose of this method, a sample taken from one of the following sources: surface, ground, storm runoff, industrial or domestic wastewater.

4. INTERFERENCES

4.1. Volatile amines can diffuse through the hydrophobic membrane to produce a conductivity response. Generally, interference from low molecular weight amines is not a practical problem because their natural occurrence is so limited.

4.2. Chloramines can decompose to produce ammonia.

4.3. Deionized water used in reagents and standards must be high quality and ammonia-free to avoid interference.

5. SAFETY

5.1. This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of any chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

6. EQUIPMENT AND SUPPLIES

6.1. Sample Containers: As per current 40 CFR Part 136 Table II, polyethylene, fluoropolymer or glass..

6.2. Class A Volumetric Flasks: various sizes used for preparation of standards.

6.3. Test Tubes: 15 x 85 mm; used in autosampler.

6.4. Analytical Balance: analytical, capable of accurately weighing to 0.0001 ± 0.00005 g.

6.5. pH METER: double junction electrode; used when adjusting the pH of the boric acid solution.

6.6. Ammonia Analyzer with gas permeation cell and conductivity detector: Timberline Instruments TL-2800 Ammonia Analyzer or an equivalent instrument that uses the same chemistry/determinative techniques and can meet the QC performance criteria set in the method.

7. REAGENTS AND STANDARDS

7.1. Reagent Water – Deionized water that does not contain measurable quantities of ammonia above the detection level of the dynamic range or any interfering volatile conductive compounds. This water will be used for all standards, calibration zero, LRB, LFB, and sample dilutions preparation.

(Note: Prolonged storage of reagent water will expose it to ammonia absorption from the laboratory environment. Prepare fresh as needed.)

7.2. Caustic Solution: Sodium Hydroxide (NaOH) or Potassium Hydroxide- 50% (w/v) solution. Prepare by weighing a known amount of ACS or better grade sodium hydroxide or potassium hydroxide to the nearest gram and add slowly to a known volume of reagent water in mL with the ratio of 1 gram sodium hydroxide or potassium hydroxide to 1 mL of reagent water. Mix thoroughly in a beaker with a magnetic stirrer.

(Caution: Reaction produces excessive heat and is caustic to unprotected skin. Prolonged storage of reagent water will expose it to ammonia absorption from the laboratory environment. Prepare fresh as needed.)

7.3. Caustic Working Solutions – 1-15% (w/v). Select caustic solution as needed per sample characteristics. Selection is based on the caustic solutions ability to raise the pH of the sample to a point that free ammonia is formed to diffuse through the membrane. Typically, with most ammonia concentrations, the 5% caustic solution is utilized. Other caustic solution concentrations may be needed based on higher ammonia concentrations, lower ammonia concentration or water matrix. Final caustic solution concentration will be based on ammonia concentration in the sample, the water matrix and QC performance requirements.

7.3.1. 1 %: Dilute 20 ml of 50% (w/w) sodium hydroxide or potassium hydroxide solution to 1 L with reagent water. Mix well by inversion. Prepare fresh weekly

7.3.2. 5 %: Dilute 100 ml of 50% (w/w) sodium hydroxide or potassium hydroxide solution to 1 L with reagent water. Mix well by inversion. Prepare fresh weekly.

7.3.3. 10%: Dilute 200 ml of 50% (w/w) sodium hydroxide or potassium hydroxide solution to 1 L with reagent water. Mix well by inversion. Prepare fresh weekly.

7.3.4. 15%: Dilute 300 ml of 50% (w/w) sodium hydroxide or potassium hydroxide solution to 1 L with reagent water. Mix well by inversion. Prepare fresh weekly.

7.4. Boric Acid Solution 10,000 ppm boric acid (H_3BO_3). Add 10 ± 0.001 g of ACS grade or better boric acid in a 1 L volumetric flask and dilute to volume with reagent water. Mix until the boric acid has completely dissolved in the solution.

(Note: Prolonged storage of Boric Acid will expose it to ammonia absorption from the laboratory environment. Prepare fresh as needed.)

7.5. Ammonium Hydroxide (NH_4OH) - 1.0 N. Transfer 135 mL of 28% ACS grade or better ammonium hydroxide to ~ 100 mL of reagent water in a 1 L volumetric flask. Dilute to the mark with reagent water and mix well. Make fresh monthly.

7.5.1. Ammonium Hydroxide Working Solutions - 0.01 N Ammonium hydroxide. Transfer 1 mL of 1 N ammonium hydroxide to 100 mL volumetric flask. Dilute to volume with reagent water. Mix well by inversion. Prepare fresh weekly.

7.6. Buffer Solutions – pH adjusted Boric acid, Select boric acid solution as needed per sample characteristics. Typically, with most ammonia concentrations, the 250 ppm Buffer Solution is utilized. Other Buffer Solution concentrations may be needed based on higher ammonia concentrations (100 ppm ammonia or greater) or lower ammonia concentration (0.1 ppm or lower).Final Buffer Solution concentration will be based on ammonia concentration in the sample and QC performance requirements.

7.6.1. 100 ppm. Transfer 10 mL of the 10,000 ppm boric acid solution to a 2 L beaker. Add ~ 975 mL of reagent water. While stirring adjust the pH to 6.5-7 using 0.01 or 0.02 N ammonium hydroxide solution. Prepare fresh weekly

7.6.2. 250 ppm. Transfer 25 mL of the 10,000 ppm boric acid solution to a 2 L beaker. Add ~ 930 mL of reagent water. While stirring adjust the pH to 6.5-7 using 0.01 or 0.02 N ammonium hydroxide solution. Prepare fresh weekly.

7.6.3. 400 ppm. Transfer 40 mL of the 10,000 ppm boric acid solution to a 2 L beaker. Add ~ 875 mL of reagent water. While stirring adjust the pH to 6.5-7 using 0.01 or 0.02 N ammonium hydroxide solution. Prepare fresh weekly.

7.7. Ammonia Standard Solutions

7.7.1. Calibration Blank: Reagent water. Replace daily or per analytical batch.

7.7.2. Ammonia Stock Solution: Accurately weigh out, to the nearest 0.001 ± 0.0005 g, dried (at 100° C) ACS grade ammonium sulfate $(NH_4)_2SO_4$ or ammonium chloride (NH_4Cl) to make the desired ammonia concentration in Table 1, below. Transfer the reagent to a 1L volumetric flask and dilute to volume with reagent water. Mix well by inversion. Prepare fresh monthly.

Table 1: Ammonia Stock Standards

Ammonium Salt	**Ammonium Chloride**	**Ammonium Sulfate**
Molecular Weight	53.49	132.14
Ammonia-N Concentration	**Ammonia Salt Weight (g)**	
2000 mg/L	7.641	9.438
500 mg/L	1.910	2.360
100 mg/L	0.382	0.472
10 mg/L	0.038	0.047

7.7.3. Calibration Standards –A minimum of 5 calibration concentrations along with a calibration blank will be required to prepare the initial calibration curve and

ongoing calibration curve. The calibration curve fit can be either linear or quadratic, but must have a R value of 0.995 or higher. Prepare the calibration standards over the dynamic range of interest as defined in Table 2 from dilutions of the ammonia stock solution. The calibration standards must be prepared using regent grade water and prepared fresh weekly.

Table 2: Membrane Dynamic Range		
Membrane Type	**Membrane Length (Inches)**	**Concentration Calibration Dynamic Range (Ammonia-N mg/L)**
High Sensitivity	10	2-0.020
Standard	4	500-0.0500

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT, DIGESTION and STORAGE

8.1. Sample Collection

8.1.1. Collect and store ammonia or Kjeldahl samples in glass or high density polyethylene bottles. Most reliable results are obtained on fresh samples.. For preservation for up to 28 d, preserve samples by acidifying to pH <2 with sulfuric acid upon collection and store at 6°C to just above 0°C, without freezing

8.2. Total Kjeldahl Nitrogen Digestion

8.2.1. Digest Kjeldahl sample as per approved method in 40 CFR Part 136 Table IB (e.g. 4500-N_{org} -1997). Digested sample is stored in a covered borosilicate container at room temperature until analyzed. Analyze within 4 hours of digestion completion.

9. QUALITY CONTROL

9.1. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. Control charts of each QC check will be kept and appropriate control limits calculated. Any QC result that fails to meet control criteria must be documented and corrective action, including a root cause analysis, must be performed.

9.2. Initial Demonstration of Capability: A laboratory fortified blank whose concentration is between the 10% and 50% of the dynamic range is analyzed four times. The mean recovery and standard deviation are calculated and evaluated for acceptance.

9.2.1. Acceptance Criteria: IDC control limit for the laboratory is based on the limits determined in this method and shall not exceed 82%-110 % with a percent Relative Standard Deviation less than 8%.

9.2.2. Corrective Action: If the IDC recovery falls outside of these limits, the analyst or instrument is judged to be out of control. A root cause analysis must be performed, corrective action taken, all findings recorded and the IDC repeated until passed.

9.3. Continuing Calibration Verification: At least one CCV standard will be prepared so as to have a concentration in the dynamic range of the instrument. The CCV will be run at least once per each batch. The CCV percent recovery is calculated and evaluated for acceptance.

9.3.1. Acceptance Criteria: CCV control limit for the laboratory is based on the limits determined in this method and shall not exceed 90%-110 %.

9.3.2. Corrective Action: If the CCV recovery falls outside of these limits, the batch is judged to be out of control. A root cause analysis must be performed, corrective action taken, all findings recorded and the sample batch repeated.

9.4. Laboratory Reagent Blank: A LRB is analyzed as a sample at least once per batch. The LRB concentration result will be evaluate for acceptance.

9.4.1. Acceptance Criteria: The LRB concentration result will be below the lowest calibration standard in the dynamic range.

9.4.2. Corrective Action: If the LRB falls outside of the acceptance limit, the batch is judged to be out of control. A root cause analysis must be performed, corrective action taken, all findings recorded and the sample batch repeated.

9.5. Laboratory Fortified Blank: A LFB with a concentration of ammonia between 10% and 50% of the dynamic range is analyzed at least once per batch. The LFB concentration result will be evaluate for acceptance.

9.5.1. Acceptance Criteria: LFB percent recovery is based on the limits determined in this method and shall not exceed 87%-104 %.

9.5.2. Corrective Action: If the LFB recovery falls outside of these limits, the batch is judged to be out of control. A root cause analysis must be performed, corrective action taken, all findings recorded and the sample batch repeated.

9.6. Laboratory Fortified Sample Matrix Spikes (LFM/LFMD): A duplicate set of ammonia or Kjeldahl samples are spiked with a known amount of ammonia with a concentration that is between 10% and 50% of the dynamic range

9.6.1. Acceptance Criteria: The LFM/LFMD percent recovery is based on the limits determined in this method and shall not exceed 84%-115 %. The relative percent difference (RPD) shall not exceed 20%.

9.6.2. Corrective Action: If the LFM and LFMD percent recovery or the RPD fall outside of these limits, the batch is judged to be out of control. A root cause analysis must be performed, corrective action taken, all findings recorded and the sample batch repeated.

9.7. Ongoing Demonstration of Capability (ODC): A LFB with a concentration of ammonia between 10% and 50% of the dynamic range is analyzed at least once per batch. The LFB percent recovery will be charted by control charts and be evaluated for acceptance.

9.7.1. Acceptance Criteria: LFB percent recovery must stay within the control limits calculated for the control chats.

9.7.2. Corrective Action: If the LFB percent recovery falls outside of these control

limits, the batch is judged to be out of control. A root cause analysis must be performed, corrective action taken, all findings recorded and the sample batch repeated.

10. PROCEDURE

10.1. Sample Analysis Sequence: Typical sample analysis sequence .

10.1.1. Instrument Start Up

10.1.2. Calibration zero

10.1.3. Calibration standards, 1-5

10.1.4. LRB

10.1.5. LFB

10.1.6. Sample used for LFM/LFMD

10.1.7. LFM

10.1.8. LFMD

10.1.9. Samples (First half of batch)

10.1.10. CCV

10.1.11. Samples (Second half of batch)

10.1.12. Repeat CCV (Optional)

10.2. Instrument Preparation

10.2.1. Start the instrument as per the Timberline TL2800 instrument manual (or an equivalent instrument that uses the same chemistry/determinative techniques and can meet the QC performance criteria set in the method.).

10.2.2. Prepare quality control samples.

10.3. Sample Preparation

10.3.1. Remove refrigerated samples from the refrigerator and warm to room temperature.

10.3.2. Kjeldahl digested samples are diluted 10:1 with the reagent water to achieve a nominal 10% (wt) sulfuric acid.

10.3.3. Spike the LFB and LFM/LFMD per QC spiking requirement.

10.3.4. Prepare CCV standard.

10.3.5. Prepare LRB water.

10.3.6. Prepare a calibration zero that is ammonia free water.

10.3.7. Prepare five (5) calibration standards that bracket the dynamic range of the analysis. The lowest concentration standard must be at or above the ML.

10.3.8. Transfer aliquots of all samples to autosampler tubes.

10.4. Initial Calibration:

10.4.1. Initial calibration is performed at the beginning of each batch.

10.4.2. Calibrate instrument with calibration zero and five calibration standards of ammonia.

10.4.3. Apply linear or polynomial curve-fitting statistics, as appropriate, to analyze the concentration–instrument response relationship.

10.4.3.1. Acceptance Criteria: The linear or nonlinear correlation coefficient for standard concentration-to-instrument response shall be greater than or equal to 0.995.

10.4.3.2. Corrective Action: If the correlation coefficient falls outside of the limit, the initial calibration is judged to be out of control. A root cause analysis must be performed, corrective action taken, all findings recorded and the initial calibration repeated.

10.4.4. Back calculate the standard concentration of each calibration point using the calibration equation determined by the curve fitting statistics.

10.4.4.1. Acceptance Criteria: The back-calculated and true concentrations should agree within ± 10% and cannot exceed ± 15%.

10.4.4.2. Corrective Action: If the standard back calculations fall outside of the limits, the initial calibration is judged to be out of control. A root cause analysis must be performed, corrective action taken, all findings recorded and the initial calibration repeated.

10.5. Continuing Calibration Verification:

10.5.1. A CCV is analyzed at the midpoint of the batch sample set. The CCV is prepared from a different source (chemical lot) than that used for the calibration standards.

10.5.1.1. Acceptance Criteria: CCV control limit for the laboratory is based on the limits determined in this method and shall not exceed 90%-110 %.

10.5.1.2. Corrective Action: If the CCV recovery falls outside of these limits, the batch is judged to be out of control. A root cause analysis must be performed, corrective action taken, all findings recorded and the sample batch repeated.

10.6. Sample Analysis

10.6.1. Operate the instrument as per manual instructions.

10.6.2. Report the values of all samples and QC samples analyzed.

10.6.3. Determine the appropriate timing parameters and integration times for the peaks.

10.6.4. Load the filled autosampler tubes into the autosampler and start the injection sequence.

10.6.5. Calibrate the system by generating a calibration curve with 5 standards and an instrument blank.

Note: (For Kjeldahl samples) The samples need to be run with a 10-15% (w/v) NaOH solution to neutralize the acid (22.5% molar excess).

10.6.6. Analyze the samples and QC checks. The sample concentration should not exceed the range of the initial calibration curve. If the sample does fall outside the range of the calibration, a new calibration curve should be run to include the range of the sample or the sample should be diluted with reagent water to a concentration within the dynamic range.

11. CALCULATIONS / DATA REDUCTION

11.1. Calculate the ammonia concentrations using the initial calibration curve generated. The batch should be reviewed for any incorrect peak identification or poor integration.

11.2. Samples and QC sample final concentrations are calculated as follow :

11.2.1. $(s \times \text{Diluted Sample Concentration}) = \text{Sample Concentration}$

$s = \text{Dilution Correction}$

11.3. Laboratory Reagent Blank (LRB); The LRB will be run at a minimum of once per sample batch. Results are reported as sample concentration and should be at or below the ML. Laboratory Fortified Blank (LFB): The LFB percent recovery is calculated as follows:

11.3.1. Percent Recovery for LFB

$$\left(\frac{\text{Experimental Value}}{\text{Expected Value}}\right) * 100 = \text{Percent Recovery LFB}$$

Experimental Value = LFB Concentration determined experimentally

Expected Value = Known LFB concentration

11.4. Initial Demonstration of Capability (IDC) is determined by calculating for four LFBs, the mean percent recovery and standard deviation.

11.4.1. $$\bar{X}_{(\text{Mean})} = \left(\sum_i X_i\right) \Big/ n$$

11.4.2. $$S_{(\text{Standard Deviation})} = \left[\frac{\sum_{i=1}^{n} (X_i - \bar{X})^2}{n-1}\right]^{\frac{1}{2}}$$

11.4.3. $$\% \text{ Relative Standard Deviation} = \left(S/\bar{x}\right) \times 100$$

11.5. Laboratory Fortified Sample Matrix -Laboratory Fortified Sample Matrix Duplicate (LFM-LFMD): The LFM/LFMD percent recoveries and RPD are calculated as follows:

11.5.1. Percent Recovery for LFM

$$\left(\frac{\text{Spiked Value} - (\text{s x Unspiked Value})}{\text{Concentration of Spike}}\right) * 100 = \text{Percent Recovery LFM}$$

Spiked Value = LFM concentration determined experimentally
Unspiked Value = Concentration of sample before spiking
s=Dilution Correction

11.5.2. Relative Percent Difference (RPD)

$$\left(\frac{(\text{LFM-LFMD})}{\left(\frac{\text{LFM+LFMD}}{2}\right)}\right) * 100 = \text{RPD}$$

LFM = Concentration determined for LFM
LFMD = Concentration determined for LFM duplicate

11.6. Continuing Calibration Verification (CCV): Check standards will be prepared so as to have a concentration between the lower and upper calibration of the dynamic range.

11.6.1. $$\left|\frac{\text{CCV Value - Initial Standard Value}}{\text{Initial Standard Value}}\right| * 100 = \text{Percent Difference}$$

11.7. Control Charts: Control charts will be kept for LFB and LFM percent recovery per sample batch, LFM and LFMD RPD per sample batch, and LRB concentration. Trends will be calculated to show whether the values determined go outside the control limits. If these trends exceed control limits of this method, then corrective action (Root Cause Analysis) must be performed.

11.8. Corrective Action (Root Cause Analysis): The laboratory analyst(s) and laboratory management will perform a root cause analysis for any QC failures. The analysis will have at a minimum the following areas described in detail:

11.8.1. Identify the problem: Identify the QC failure. Include instrument, reagent, sampling, personnel and any other problems.

11.8.2. Investigate to identify the root cause: Determine how each problem identified interacted with each other to create the QC problem.

11.8.3. Come up with the solution: Develop an encompassing solution to address all problems that created the QC failure.

11.8.4. Implement the solution: Develop an implementation plan that includes all components of the developed solution and have laboratory management implement it.

11.8.5. Document the solution: Document all corrective action steps taken under laboratory management implementation of the corrective action.

11.8.6. Communicate the solution: Develop training and management programs to communicate and evaluate all personnel included in the corrective action solution.

11.8.7. Evaluate the effectiveness of the solution: Document QC results in trend charts and laboratory staff performance to validate corrective action solution.

12. METHOD PERFORMANCE

12.1. Method Detection Limit and Method Limit Study(MDL-ML):

12.1.1. MDL and ML are determined for the initial start-up of the instrument prior to any sample analysis and when the instrument has had maintenance or repairs of the membrane, detector or any other internal analytical components. These requirements do not include consumable supplies or reagents. Analytical procedures for the MDL and ML are listed in the current promulgated 40 CFR 136 Appendix B.

12.1.2. Prepare seven samples at three to five times the estimated MDL concentration. The MDL samples are prepared in the Synthetic

12.1.3. Prepare and analyze the MDL standards as described in Section 10.

12.1.4. Calculate the average concentration found in µg/L or mg/L, and the standard deviation of the concentration(s) for each analyte .

12.1.5. The MDL is calculated as Students t for the 99th percentile times the standard deviation of the MDL replicate results, following the procedure at 40 CFR 136, Appendix B.

12.1.6. The ML is calculated as per "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; National Primary Drinking Water Regulations; and National Secondary Drinking Water Regulations: Analysis and Sampling Procedures".

12.1.7. $(\text{MDL} \times 3.18) = \text{ML}$

12.2. Instrument Detection Limit Study:

12.2.1. Prepare three laboratory reagent blanks.

12.2.2. Analyze the IDL blanks as per Section 10.

12.2.3. The IDL is calculated as per Standard Methods for the Examination of Water and Wastewater Part 1000.

12.2.4. Instrument Detection Limit = $(3 \times S_{LRB})$

S_{LRB} = Standard Deviation of Laboratory Reagent Blanks

12.3. Demonstration of Capabilities

12.3.1. All laboratory personnel are required to perform an initial demonstration of capability (IDC) on the instrument they will be using for analysis prior to testing samples. Ongoing demonstration of capability must be demonstrated by

control charts with LFBs being analyzed at least once per sample batch. Initial and ongoing demonstrations of capabilities are conducted as follows.

12.3.2. Initial Demonstration of Capability Analyst Proficiency - Four LFBs are analyzed using the same instrumental conditions and procedures used to analyze samples. Using these four LFBs demonstrates the analyst's ability to optimize and calibrate the instrument and to prepare analytical solutions. Calculate the average percent recovery and standard deviation of the recovery.

12.3.2.1. Acceptance Criteria: IDC control limit for the laboratory is based on the limits determined in this method and shall not exceed 82%-110 % with a percent Relative Standard Deviation less than 8%.

12.3.2.2. Corrective Action: If the IDC recovery falls outside of these limits, the analyst or instrument is judged to be out of control. A root cause analysis must be performed, corrective action taken, all findings recorded and the IDC repeated until passed.

12.3.3. Ongoing Demonstration of Capability Analyst Proficiency: A LFB is analyzed using the same instrumental conditions and procedures used to analyze samples at least once per batch. The LFB percent recovery will be charted by control charts and be evaluated for acceptance.

12.3.3.1. Acceptance Criteria: LFB percent recovery must stay within the control limits calculated for the control chats.

12.3.3.2. Corrective Action: If the LFB percent recovery falls outside of these control limits, the batch is judged to be out of control. A root cause analysis must be performed, corrective action taken, all findings recorded and the sample batch repeated.

13. POLLUTION CONTROL

13.1. It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect air, water, and land by minimizing and control all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

14. WASTE MANAGEMENT

14.1. There are no standards or reagents used in this method at the concentrations required that pose a threat to the environment. Refer to Local, State or Federal for correct disposal of all chemicals.

15. REFERENCES / BIBLIOGRAPHY

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16. ATTACHMENTS

Note: All concentrations are for Ammonia-N

16.1. Table A: General Instrument Parameters

16.2. Table B: 4 inch Membrane Pooled MDLs for Individual Laboratories and All Laboratories

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16.12. Table L: 10 Inch Membrane Calibration Verification

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16.18. Table R: Single Operator Standard Deviation and %RSD – 4 Inch Membrane

16.19. Table S:Youden Pair Recovery - 10 Inch Membrane

16.20. Table T: Single Operator Standard Deviation and %RSD – 10 Inch Membrane

16.21. Table U: Laboratory Reagent Blank – Instrument Detection Limit 4 Inch Membrane

16.22. Table V: Laboratory Reagent Blank – Instrument Detection Limit 10 Inch Membrane

Table A: General Instrument Parameters												
Lab	1	2	3	4	5	6	7	8	9	10	9	6
Timberline Instrument Model #	TL-200	2800	TL-2800	TL-201	TL-2800 / SN 121209001	TL 2800	TL2100	TL 2800	TL-2800	TL-2100	TL-2800	TL 2800
Membrane Length (Inches)	4 Inches	4 Inches	4 Inches	4 Inches	4 Inches	4 Inches	4 Inches	4 Inches	4 Inches	10 Inches	10 Inches	10 inch
Gain (# or Letter)	C	1	C	10	1	1000	D	10	10	10	100	1000
Attenuation (# or Letter)	2	2	1	4	div 4	div 8	N/A	div 8	div 4	div 1	div 4	div 2
Buffer Concentration and pH	150 ppm Boric Acid pH 7.2	.1g boric acid per liter of water pH = 7.02	0.1 g/L Boric Acid at pH of 7.0	pH=7.2 | 28-30% NH3OH=3.0mL/L 3.75 ml of that Liter + 0.1g Boric Acid	250 ppm / pH = 6.5	250 ppm pH 6.51	Conc = 0.01% PH = 6.99	250 ppm, pH 6.9	400 ppm boric acid, pH 6.0	100 mg/L, pH 6.5	400ppm boric acid, pH 6.0	400 ppm pH 6.51
Caustic Concentration	0.05	10g DTPA and 25g potassium hydroxide per liter of water	25 g/L Potassium Hydroxide and 10 g/L of DTPA	0.97N NaOH (38.9g) 11.1g/L DTPA	5% NaOH	0.05	5 % KOH + 2 % DTPA	15% NaOH	5% KOH	0.10%	1% KOH	1%
Injection Time (seconds)	15	20	15	20 seconds	10 s	20	24 seconds	15 sec	25	20	25	20 sec
Pump speed (rpm)	45	29	40	50	28 rpm	30	45 (rpm)	28	40	28	40	30
Sample Matrix Type	40 CFR 442 Commercial Truck Wash	Raw POTW Influent	Final POTW Effluent Dechlorinated	40 CFR 430 Paper Pulp Mill Effluent	POTW Return Waste Activated Sludge Centrifugation Liquor	40 CFR 433 Metal Finisher Effluent	POTW Primary Clarifier Effluent	TKN Waste Water Manure Lagoon	POTW Effluent, Non-Disinfected	POTW Receiving Watershed	POTW Effluent Non-Disinfected.	40 CFR 433 Metal Finisher Effluent

Table B: 4 inch Membrane Pooled MDLs for Individual Laboratories and All Laboratories																		
Lab																		
1	Standard or Sample	Sample Concentration (PPB N as Ammonia)	Measurement 1	Measurement 2	Measurement 3	Average Measurement 1	Measurement 1 Calc 1	Average Measurement 2	Measurement 2 Calc 1	Average Measurement 3	Measurement 3 Calc 1	Sum N Individual Laboratories	Individual Laboratory Pooled Standard Deviation	Individual Laboratories Student T	Individual Laboratory Pooled MDL	Total Laboratories MDL	Total Laboratories ML	Reporting ML
	MDL Sample 1	50	49.8	48.9	49.3	42.1	268.116366	40.9	262.1394838	40.5	260.1670487	18	6.626641929	2.552	17	14	46	5 E+01
	MDL Sample 2	50	51.6	47.9	46.6	42.1		40.9		40.5								
	MDL Sample 3	50	45.0	45.5	44.9	42.1		40.9		40.5								
	MDL Sample 4	50	39.5	39.3	39.0	42.1		40.9		40.5								
	MDL Sample 5	50	35.7	32.7	31.5	42.1		40.9		40.5								
	MDL Sample 6	50	37.2	35.7	36.6	42.1		40.9		40.5								
	MDL Sample 7	50	36.0	36.2	35.4	42.1		40.9		40.5								
2	Standard or Sample	Sample Concentration (PPB N as Ammonia)	Measurement 1	Measurement 2	Measurement 3	Average Measurement 1	Measurement 1 Calc 1	Average Measurement 2	Measurement 2 Calc 1	Average Measurement 3	Measurement 3 Calc 1	Sum N Individual Laboratories	Individual Laboratory Pooled Standard Deviation	Individual Laboratories Student T	Individual Laboratory Pooled MDL			
	MDL Sample 1	50	46.3	45.1	34.0	46.4	22.10172622	48.8	278.643543	42.8	609.387987	18	7.110763579	2.552	18			
	MDL Sample 2	50	46.2	44.0	33.1	46.4		48.8		42.8								
	MDL Sample 3	50	44.4	40.9	34.4	46.4		48.8		42.8								
	MDL Sample 4	50	44.0	47.6	40.8	46.4		48.8		42.8								
	MDL Sample 5	50	46.4	50.3	45.4	46.4		48.8		42.8								
	MDL Sample 6	50	49.5	56.0	54.6	46.4		48.8		42.8								
	MDL Sample 7	50	48.2	58.0	57.5	46.4		48.8		42.8								

	Standard or Sample	Sample Concentration (PPB N as Ammonia)	Measurement 1	Measurement 2	Measurement 3	Average Measurement 1	Measurement 1 Calc 1	Average Measurement 2	Measurement 2 Calc 1	Average Measurement 3	Measurement 3 Calc 1	Sum N Individual Laboratories	Individual Laboratory Pooled Standard Deviation	Individual Laboratories Student T	Individual Laboratory Pooled MDL
3	MDL Sample 1	50	36.5	39.5	29.7	47.8	1000.816948	42.3	2200.645448	55.7	1363.948179	18	15.92588838	2.552	41
	MDL Sample 2	50	39.2	28.9	39.6	47.8		42.3		55.7					
	MDL Sample 3	50	63.7	39.2	66.8	47.8		42.3		55.7					
	MDL Sample 4	50	49.3	38.5	63.6	47.8		42.3		55.7					
	MDL Sample 5	50	33.6	73.0	67.9	47.8		42.3		55.7					
	MDL Sample 6	50	66.3	18.4	57.2	47.8		42.3		55.7					
	MDL Sample 7	50	46.0	58.5	65.1	47.8		42.3		55.7					
	Standard or Sample	Sample Concentration (PPB N as Ammonia)	Measurement 1	Measurement 2	Measurement 3	Average Measurement 1	Measurement 1 Calc 1	Average Measurement 2	Measurement 2 Calc 1	Average Measurement 3	Measurement 3 Calc 1	Sum N Individual Laboratories	Individual Laboratory Pooled Standard Deviation	Individual Laboratories Student T	Individual Laboratory Pooled MDL
4	MDL Sample 1	50	33.4	36.2	38.2	35.3	295.9283579	36.0	442.95748	35.5	175.0779986	18	7.125711802	2.552	18
	MDL Sample 2	50	29.3	22.5	33.0	35.3		36.0		35.5					
	MDL Sample 3	50	31.0	38.3	37.3	35.3		36.0		35.5					
	MDL Sample 4	50	37.7	36.2	31.9	35.3		36.0		35.5					
	MDL Sample 5	50	30.9	30.6	36.0	35.3		36.0		35.5					
	MDL Sample 6	50	49.9	50.8	44.6	35.3		36.0		35.5					
	MDL Sample 7	50	34.6	37.5	27.6	35.3		36.0		35.5					

	Standard or Sample	Sample Concentration (PPB N as Ammonia)	Measurement 1	Measurement 2	Measurement 3	Average Measurement 1	Measurement 1 Calc 1	Average Measurement 2	Measurement 2 Calc 1	Average Measurement 3	Measurement 3 Calc 1	Sum N Individual Laboratories	Individual Laboratory Pooled Standard Deviation	Individual Laboratories Student T	Individual Laboratory Pooled MDL
5	MDL Sample 1	50	53.8	52.5	50.2	49.8	67.595018	50.5	54.274461	49.8	40.76319743	18	3.005852407	2.552	8
	MDL Sample 2	50	53.9	50.3	53.6	49.8		50.5		49.8					
	MDL Sample 3	50	50.1	51.3	50.4	49.8		50.5		49.8					
	MDL Sample 4	50	51.2	55.5	50.2	49.8		50.5		49.8					
	MDL Sample 5	50	48.0	48.5	47.4	49.8		50.5		49.8					
	MDL Sample 6	50	46.4	47.2	45.6	49.8		50.5		49.8					
	MDL Sample 7	50	45.6	47.9	51.6	49.8		50.5		49.8					
	Standard or Sample	Sample Concentration (PPB N as Ammonia)	Measurement 1	Measurement 2	Measurement 3	Average Measurement 1	Measurement 1 Calc 1	Average Measurement 2	Measurement 2 Calc 1	Average Measurement 3	Measurement 3 Calc 1	Sum N Individual Laboratories	Individual Laboratory Pooled Standard Deviation	Individual Laboratories Student T	Individual Laboratory Pooled MDL
6	MDL Sample 1	50	52.5	53.8	52.8	50.9	13.4269972	50.1	33.26565203	49.8	18.15411917	18	1.89805117	2.552	5
	MDL Sample 2	50	50.0	48.2	49.3	50.9		50.1		49.8					
	MDL Sample 3	50	50.1	50.4	49.4	50.9		50.1		49.8					
	MDL Sample 4	50	50.3	49.5	48.2	50.9		50.1		49.8					
	MDL Sample 5	50	51.1	49.8	49.7	50.9		50.1		49.8					
	MDL Sample 6	50	53.4	51.7	51.3	50.9		50.1		49.8					
	MDL Sample 7	50	49.2	47.3	47.7	50.9		50.1		49.8					

	Standard or Sample	Sample Concentration (PPB N as Ammonia)	Measurement 1	Measurement 2	Measurement 3	Average Measurement 1	Measurement 1 Calc 1	Average Measurement 2	Measurement 2 Calc 1	Average Measurement 3	Measurement 3 Calc 1	Sum N Individual Laboratories	Individual Laboratory Pooled Standard Deviation	Individual Laboratories Student T	Individual Laboratory Pooled MDL
7	MDL Sample 1	50	52.2	53.4	48.7	52.9	56.08613904	53.6	23.69668369	52.1	47.21292083	18	2.656185063	2.552	7
	MDL Sample 2	50	54.6	50.3	48.4	52.9		53.6		52.1					
	MDL Sample 3	50	46.7	54.2	50.9	52.9		53.6		52.1					
	MDL Sample 4	50	52.6	54.5	54.0	52.9		53.6		52.1					
	MDL Sample 5	50	56.2	52.3	52.5	52.9		53.6		52.1					
	MDL Sample 6	50	53.3	55.9	54.7	52.9		53.6		52.1					
	MDL Sample 7	50	54.7	54.6	55.3	52.9		53.6		52.1					
	Standard or Sample	Sample Concentration (PPB N as Ammonia)	Measurement 1	Measurement 2	Measurement 3	Average Measurement 1	Measurement 1 Calc 1	Average Measurement 2	Measurement 2 Calc 1	Average Measurement 3	Measurement 3 Calc 1	Sum N Individual Laboratories	Individual Laboratory Pooled Standard Deviation	Individual Laboratories Student T	Individual Laboratory Pooled MDL
8	MDL Sample 1	50	50.4	30.6	48.7	48.0	86.49537546	41.3	609.6544895	47.3	17.89875795	17	6.480961362	2.567	17
	MDL Sample 2	50	†	50.6	44.3	48.0		41.3		47.3					
	MDL Sample 3	50	44.1	49.2	48.0	48.0		41.3		47.3					
	MDL Sample 4	50	53.1	39.1	48.7	48.0		41.3		47.3					
	MDL Sample 5	50	42.0	35.9	45.6	48.0		41.3		47.3					
	MDL Sample 6	50	49.9	42.8	47.4	48.0		41.3		47.3					
	MDL Sample 7	50	48.5	41.0	48.6	48.0		41.3		47.3					

	Standard or Sample	Sample Concentration (PPB N as Ammonia)	Measurement 1	Measurement 2	Measurement 3	Average Measurement 1	Measurement 1 Calc 1	Average Measurement 2	Measurement 2 Calc 1	Average Measurement 3	Measurement 3 Calc 1	Sum N Individual Laboratories	Individual Laboratory Pooled Standard Deviation	Individual Laboratories Student T	Individual Laboratory Pooled MDL
9	MDL Sample 1	50	52.4	48.9	51.1	47.7	71.10356752	46.0	148.0760933	45.5	157.3451706	18	4.57362506	2.552	12
	MDL Sample 2	50	46.6	48.0	48.2	47.7		46.0		45.5					
	MDL Sample 3	50	49.9	54.2	46.9	47.7		46.0		45.5					
	MDL Sample 4	50	45.8	42.3	40.5	47.7		46.0		45.5					
	MDL Sample 5	50	43.5	42.4	38.9	47.7		46.0		45.5					
	MDL Sample 6	50	44.5	43.7	51.3	47.7		46.0		45.5					
	MDL Sample 7	50	51.1	42.1	41.8	47.7		46.0		45.5					

†: Data value determined to be an outlier

Table C: 10 inch Membrane Pooled MDLs for Individual Laboratories and All Laboratories																		
Lab																		
10	Standard or Sample	Sample Concentration (PPB N as Ammonia)	Measurement 1	Measurement 2	Measurement 3	Average Measurement 1	Measurement 1 Calc 1	Average Measurement 2	Measurement 2 Calc 1	Average Measurement 3	Measurement 3 Calc 1	Sum N Individual Laboratories	Individual Laboratory Pooled Standard Deviation	Individual Laboratories Student T	Individual Laboratory Pooled MDL	Total Laboratories MDL	Total Laboratories ML	Reporting ML
	MDL Sample 1	20	20.2	20.4	20.7	19.1	10.22363599	19.3	4.685146617	19.4	4.310019405	18	1.033301129	2.552	3	2	7	1.E+01
	MDL Sample 2	20	18.4	18.3	20.0	19.1		19.3		19.4								
	MDL Sample 3	20	21.3	20.6	20.0	19.1		19.3		19.4								
	MDL Sample 4	20	18.9	18.8	18.5	19.1		19.3		19.4								
	MDL Sample 5	20	18.8	19.0	19.3	19.1		19.3		19.4								
	MDL Sample 6	20	19.1	19.2	18.8	19.1		19.3		19.4								
	MDL Sample 7	20	17.3	18.8	18.5	19.1		19.3		19.4								
9	Sample Concentration (PPB N as Ammonia)	Measurement 1	Measurement 2	Measurement 3	Average Measurement 1	Measurement 1 Calc 1	Average Measurement 2	Measurement 2 Calc 1	Average Measurement 3	Measurement 3 Calc 1	Sum N Individual Laboratories	Individual Laboratory Pooled Standard Deviation	Individual Laboratories Student T	Individual Laboratory Pooled MDL	Sample Concentration (PPB N as Ammonia)			
	MDL Sample 1	20	18.1	23.0	20.5	20.0	14.00052526	20.5	16.54387347	19.7	14.37793392	18	1.579773764	2.552	4			
	MDL Sample 2	20	22.5	20.5	19.6	20.0		20.5		19.7								
	MDL Sample 3	20	20.8	21.5	22.5	20.0		20.5		19.7								
	MDL Sample 4	20	20.5	18.2	18.3	20.0		20.5		19.7								

	MDL Sample 5	20	20.2	18.9	20.3	20.0		20.5		19.7					
	MDL Sample 6	20	18.3	20.9	18.7	20.0		20.5		19.7					
	MDL Sample 7	20	19.8	20.6	18.1	20.0		20.5		19.7					
	Sample Concentration (PPB N as Ammonia)	Measurement 1	Measurement 2	Measurement 3	Average Measurement 1	Measurement 1 Calc 1	Average Measurement 2	Measurement 2 Calc 1	Average Measurement 3	Measurement 3 Calc 1	Sum N Individual Laboratories	Individual Laboratory Pooled Standard Deviation	Individual Laboratories Student T	Individual Laboratory Pooled MDL	Sample Concentration (PPB N as Ammonia)
6	MDL Sample 1	20	23.1	23.1	23.0	20.4	13.27821238	20.2	14.44861109	20.1	16.10179668	18	1.56042409	2.552	4
	MDL Sample 2	20	20.0	19.2	18.1	20.4		20.2		20.1					
	MDL Sample 3	20	20.1	18.8	19.7	20.4		20.2		20.1					
	MDL Sample 4	20	20.7	20.6	18.5	20.4		20.2		20.1					
	MDL Sample 5	20	20.1	19.4	20.7	20.4		20.2		20.1					
	MDL Sample 6	20	20.7	19.0	20.7	20.4		20.2		20.1					
	MDL Sample 7	20	18.1	21.0	19.8	20.4		20.2		20.1					

Table D: LFM/LFMD Recoveries				
4 Inch Membrane		Matrix Spike Recovery	Matrix Spike Duplicate Recovery	Relative Percent Difference
1	40 CFR 442, Commercial Truck Wash	114.00%	125.04%	9.24%
2	Raw POTW Influent	111.77%	91.80%	19.61%
3	Final POTW Effluent, Dechlorinated	132.73%	95.99%	32.12%
4	40 CFR 430, Paper Pulp Mill Effluent	96.06%	98.83%	2.85%
5	POTW Return Waste Activated Sludge Centrifugation Liquor	111.96%	111.05%	0.82%
6	40 CFR 433, Metal Finisher Effluent	115.96%	118.08%	1.81%
7	POTW Primary Clarifier Effluent	100.06%	98.23%	1.85%
8	Manure Lagoon Waste Water (TKN)	118.38%	119.13%	0.64%
9	POTW Effluent, Non Disinfected.	102.92%	111.82%	8.28%
10 Inch Membrane		Matrix Spike Recovery	Matrix Spike Duplicate Recovery	Relative Percent Difference
10	POTW Receiving Watershed	101.12%	107.96%	6.54%
6	40 CFR 433, Metal Finisher Effluent	100.15%	92.63%	7.80%
9	POTW Effluent, Non Disinfected.	106.27%	98.58%	7.50%

Table E: 4 Inch Membrane LFM/LFMD Calculations												
	LFM-LFMD Percent Recovery					MS/MSD Calculation						
Lab	Matrix Spike Recovery	Matrix Spike Duplicate Recovery	Individual Laboratory Mean	Individual Laboratory Standard Deviation	Individual Laboratory Relative Percent Difference (RPD)	All Laboratory Mean	Pooled Within Laboratory Standard Deviation (sw)	Between Laboratory Standard Deviation of the Mean Results (sb)	RSD IPR(max)	IPR Combined Standard Deviation (sc)	All Laboratory MS/MSD Lower Limit	All Laboratory MS/MSD Upper Limit
1	114.00%	125.04%	119.52%	7.81%	9.24%	109.66%	10.45%	8.56%	30.50%	11.67%	83.99%	135.32%
2	111.77%	91.80%	101.79%	14.12%	19.61%							
3	132.73%	95.99%	114.36%	25.98%	32.12%							
4	96.06%	98.83%	97.44%	1.96%	2.85%							
5	111.96%	111.05%	111.51%	0.65%	0.82%							
6	115.96%	118.08%	117.02%	1.50%	1.81%							
7	100.06%	98.23%	99.14%	1.29%	1.85%							
8	118.38%	119.13%	118.76%	0.53%	0.64%							
9	102.92%	111.82%	107.37%	6.29%	8.28%							

Table F:10 Inch Membrane LFM/LFMD Calculations												
	LFM-LFMD Percent Recovery					MS/MSD Calculation						
Lab	Matrix Spike Recovery	Matrix Spike Duplicate Recovery	Individual Laboratory Mean	Individual Laboratory Standard Deviation	Individual Laboratory Relative Percent Difference (RPD)	All Laboratory Mean	Pooled Within Laboratory Standard Deviation (sw)	Between Laboratory Standard Deviation of the Mean Results (sb)	RPD (max)	IPR Combined Standard Deviation (sc)	All Laboratory MS/MSD Lower Limit	All Laboratory MS/MSD Upper Limit
10	101.12%	107.96%	104.54%	4.84%	6.54%	101.12%	5.20%	4.23%	23.15%	6.11%	87.67%	114.57%
6	100.15%	92.63%	96.39%	5.32%	7.80%							
9	106.27%	98.58%	102.43%	5.43%	7.50%							

Table G 1: 4 inch Membrane - LFB Overall Percent Recovery																				
LFB Recovery												Percent Recovery								
Lab	IPR Reading 1	IPR Reading 2	IPR Reading 3	IPR Reading 4	OPR Reading 1	OPR Reading 2	OPR Reading 3	OPR Reading 4	OPR Reading 5	Standard Value (PPB N)	N	IPR Reading 1	IPR Reading 2	IPR Reading 3	IPR Reading 4	OPR Reading 1	OPR Reading 2	OPR Reading 3	OPR Reading 4	OPR Reading 5
1	985.75	1018.92	1025.69	1029.08	852.95	886.68	1039.77	898.00		1000.00	8	98.58%	101.89%	102.57%	102.91%	85.30%	88.67%	103.98%	89.80%	
2	933.71	865.26	839.73	990.04	814.57	790.78	815.64	1043.32	1038.35	1000.00	9	93.37%	86.53%	83.97%	99.00%	81.46%	79.08%	81.56%	104.33%	103.83%
3	973.94	1041.90	1057.47	952.63	933.53	1014.73	964.00	1009.15		1000.00	8	97.39%	104.19%	105.75%	95.26%	93.35%	101.47%	96.40%	100.92%	
4	1053.00	1062.00	1046.00	1046.00	1040.00	1030.00	1000.00	1000.00		1000.00	8	105.30%	106.20%	104.60%	104.60%	104.00%	103.00%	100.00%	100.00%	
5	990.56	973.42	956.67	974.06	942.77	940.92	940.13	931.70		1000.00	8	99.06%	97.34%	95.67%	97.41%	94.28%	94.09%	94.01%	93.17%	
6	1022.21	1069.33	990.91	987.07	1016.58	1018.54	1027.27	1035.52		1000.00	8	102.22%	106.93%	99.09%	98.71%	101.66%	101.85%	102.73%	103.55%	
7	914.39	904.69	899.01	884.62	954.38	999.53	891.61	912.52		1000.00	8	91.44%	90.47%	89.90%	88.46%	95.44%	99.95%	89.16%	91.25%	
8	951.92	977.21	1021.87	947.83	921.95	961.28	857.54	902.88		1000.00	8	95.19%	97.72%	102.19%	94.78%	92.20%	96.13%	85.75%	90.29%	
9	911.86	909.63	892.98	889.47	879.27	851.10	852.64	841.43		1000.00	8	91.19%	90.96%	89.30%	88.95%	87.93%	85.11%	85.26%	84.14%	

Table G 2: 4 inch membrane - LFB Overall Percent Recovery				
Lab	Single Lab Average % Recovery	Single Lab Standard Deviation	Total Lab Average % Recovery	Total Lab STANDARD DEVIATION
1	96.71%	0.08	95.67%	0.07
2	90.35%	0.10		
3	99.34%	0.04		
4	103.46%	0.02		
5	95.63%	0.02		
6	102.09%	0.03		
7	92.01%	0.04		
8	94.28%	0.05		
9	87.85%	0.03		

Table H 1: 10 inch Membrane - LFB Overall Percent Recovery																		
LFB recovery											Percent Recovery							
Lab	IPR Reading 1	IPR Reading 2	IPR Reading 3	IPR Reading 4	OPR Reading 1	OPR Reading 2	OPR Reading 3	OPR Reading 4	Standard Value (PPB N)	N	IPR Reading 1	IPR Reading 2	IPR Reading 3	IPR Reading 4	OPR Reading 1	OPR Reading 2	OPR Reading 3	OPR Reading 4
10	48.48	48.05	46.91	46.79	47.78	46.51	47.60	49.04	50.00	8	96.96%	96.10%	93.81%	93.59%	95.56%	93.02%	95.19%	98.07%
6	51.33	48.78	46.65	47.49	48.84	50.28	48.13	49.09	50.00	8	102.66%	97.55%	93.30%	94.97%	97.68%	100.56%	96.26%	98.19%
9	51.38	49.48	46.51	47.76	51.17	46.04	44.42	43.61	50.00	8	102.76%	98.97%	93.03%	95.51%	102.34%	92.09%	88.85%	87.22%

Table H 2: 10 Inch Membrane - LFB Overall Percent Recovery			
Single Lab Average % Recovery	Single Lab Standard Deviation	Total Lab Average % Recovery	Total Lab Standard Deviation
95.29%	0.02	96.01%	0.04
97.65%	0.03		
95.10%	0.06		

Table I 1: 4 Inch Membrane LFB Bias and Precision																				
	LFB Recovery											Difference								
Lab	IPR Reading 1	IPR Reading 2	IPR Reading 3	IPR Reading 4	OPR Reading 1	OPR Reading 2	OPR Reading 3	OPR Reading 4	OPR Reading 5	Standard Value (PPB N)	N	IPR Reading 1	IPR Reading 2	IPR Reading 3	IPR Reading 4	OPR Reading 1	OPR Reading 2	OPR Reading 3	OPR Reading 4	OPR Reading 5
1	985.75	1018.92	1025.69	1029.08	852.95	886.68	1039.77	898.00		1000.00	8	-14.25	18.92	25.69	29.08	-147.05	-113.32	39.77	-102.00	
2	933.71	865.26	839.73	990.04	814.57	790.78	815.64	1043.32	1038.35	1000.00	9	-66.29	-134.74	-160.27	-9.96	-185.43	-209.22	-184.36	43.32	38.35
3	973.94	1041.90	1057.47	952.63	933.53	1014.73	964.00	1009.15		1000.00	8	-26.06	41.90	57.47	-47.37	-66.47	14.73	-36.00	9.15	
4c	1053.00	1062.00	1046.00	1046.00	1040.00	1030.00	1000.00	1000.00		1000.00	8	53.00	62.00	46.00	46.00	40.00	30.00	0.00	0.00	
5	990.56	973.42	956.67	974.06	942.77	940.92	940.13	931.70		1000.00	8	-9.44	-26.58	-43.33	-25.94	-57.23	-59.09	-59.87	-68.30	
6	1022.21	1069.33	990.91	987.07	1016.58	1018.54	1027.27	1035.52		1000.00	8	22.21	69.33	-9.09	-12.93	16.58	18.54	27.27	35.52	
7	914.39	904.69	899.01	884.62	954.38	999.53	891.61	912.52		1000.00	8	-85.61	-95.31	-100.99	-115.38	-45.62	-0.47	-108.39	-87.48	
8	951.92	977.21	1021.87	947.83	921.95	961.28	857.54	902.88		1000.00	8	-48.08	-22.79	21.87	-52.17	-78.05	-38.72	-142.46	-97.12	
9	911.86	909.63	892.98	889.47	879.27	851.10	852.64	841.43		1000.00	8	-88.14	-90.37	-107.02	-110.53	-120.73	-148.90	-147.36	-158.57	

Table I 2: 4 Inch Membrane LFB Bias and Precision									
LFB Squared Difference									
Lab	IPR Reading 1	IPR Reading 2	IPR Reading 3	IPR Reading 4	OPR Reading 1	OPR Reading 2	OPR Reading 3	OPR Reading 4	OPR Reading 5
1	203.0625	357.9664	659.9761	845.6464	21623.7025	12841.4224	1581.6529	10404.0000	
2	4394.2315	18155.9455	25687.1140	99.1817	34383.9140	43773.4268	33988.6096	1876.5358	1470.6458
3	678.9464	1755.7022	3302.6630	2243.7085	4418.2077	216.9140	1296.0144	83.8049	
4c	2809.0000	3844.0000	2116.0000	2116.0000	1600.0000	900.0000	0.0000	0.0000	
5	89.1891	706.7091	1877.4022	672.7280	3274.8151	3491.0372	3584.2972	4665.5730	
6	493.2467	4806.1822	82.6159	167.2118	275.0603	343.8976	743.8574	1261.6790	
7	7329.6234	9084.5813	10198.1459	13311.9767	2081.5138	0.2174	11748.0453	7653.3925	
8	2311.8787	519.2018	478.1220	2721.2916	6091.6464	1499.0835	20293.7119	9431.7117	
9	7769.3261	8165.9240	11453.9940	12216.1353	14575.5897	22171.7288	21716.3306	25144.3472	

Table I 3: 4 Inch Membrane LFB Bias and Precision				
LFB Bias and Precision				
Lab	Single Lab Bias (PPB)	Single Lab Precision (PPB)	Total Lab Bias (PPB)	Total Lab Precision (PPB)
1	-33	83	-43	88
2	-97	143		
3	-7	45		
4c	35	44		
5	-44	51		
6	21	34		
7	-80	94		
8	-57	79		
9	-121	133		

Table J 1: 10 Inch Membrane LFB Bias and Precision																		
LFB Recovery											Difference							
Lab	IPR Reading 1	IPR Reading 2	IPR Reading 3	IPR Reading 4	OPR Reading 1	OPR Reading 2	OPR Reading 3	OPR Reading 4	Standard Value (PPB N)	N	IPR Reading 1	IPR Reading 2	IPR Reading 3	IPR Reading 4	OPR Reading 1	OPR Reading 2	OPR Reading 3	OPR Reading 4
10	48.48	48.05	46.91	46.79	47.78	46.51	47.60	49.04	50.00	8	-1.52	-1.95	-3.09	-3.21	-2.22	-3.49	-2.40	-0.96
6	51.33	48.78	46.65	47.49	48.84	50.28	48.13	49.09	50.00	8	1.33	-1.22	-3.35	-2.51	-1.16	0.28	-1.87	-0.91
9	51.38	49.48	46.51	47.76	51.17	46.04	44.42	43.61	50.00	8	1.38	-0.52	-3.49	-2.24	1.17	-3.96	-5.58	-6.39

Table J 2: 10 Inch Membrane LFB Bias and Precision								
LFB Squared Difference								
Lab	IPR Reading 1	IPR Reading 2	IPR Reading 3	IPR Reading 4	OPR Reading 1	OPR Reading 2	OPR Reading 3	OPR Reading 4
10	2.3036	3.7950	9.5652	10.2801	4.9356	12.1640	5.7762	0.9306
6	1.7654	1.4962	11.2075	6.3225	1.3452	0.0785	3.5004	0.8230
9	1.9093	0.2660	12.1602	5.0388	1.3731	15.6511	31.0815	40.8576

Table J 3: 10 Inch Membrane LFB Bias and Precision				
Bias and Precision				
Lab	Single Lab Bias (PPB)	Single Lab Precision (PPB)	Total Lab Bias (PPB)	Total Lab Precision (PPB)
10	-2	3	-2	3
6	-1	2		
9	-2	4		

Table K 1: 4 Inch Membrane Calibration Verification															
Lab	Standards Response						Calibration factor						Single Laboratory		
STDS	500	1000	2000	4000	5000		500	1000	2000	4000	5000		Average	Standard Deviation	% RSD
1	0.01692	0.03551	0.07341	0.14591	0.18247		3.38E-05	3.55E-05	3.67E-05	3.65E-05	3.65E-05		3.58053E-05	1.19219E-06	3.33%
STDS	1000	2000	3000	4000	6000		1000	2000	3000	4000	6000		Average	Standard Deviation	% RSD
2	0.237583	0.497794	0.75085	0.993456	1.466973		0.000238	0.000249	0.00025	0.000248	0.000244		0.000245925	5.13344E-06	2.09%
STDS	1000	2000	4000	6000	10000		1000	2000	4000	6000	10000		Average	Standard Deviation	% RSD
3	0.043165	0.0861	0.168219	0.255619	0.396178		4.32E-05	4.31E-05	4.21E-05	4.26E-05	3.96E-05		4.20981E-05	1.45372E-06	3.45%
STDS	500	1000	2000	5000	10000		500	1000	2000	5000	10000		Average	Standard Deviation	% RSD
4	0.05077	0.113035	0.203516	0.489053	0.915959		0.000102	0.000113	0.000102	9.78E-05	9.16E-05		0.000101148	7.81357E-06	7.72%
STDS	500	2000	5000	10000	50000		500	2000	5000	10000	50000		Average	Standard Deviation	% RSD

5	0.004156	0.016651	0.041184	0.085338	0.402447		8.31E-06	8.33E-06	8.24E-06	8.53E-06	8.05E-06		8.29141E-06	1.74723E-07	2.11%
STDS	200	500	1000	5000	10000	12000	200	500	1000	5000	10000	12000	Average	Standard Deviation	% RSD
6	0.008189	0.019293	0.039304	0.191422	0.384201	0.448755	4.08E-05	3.86E-05	3.93E-05	3.83E-05	3.84E-05	3.73963E-05	3.88069E-05	1.1705E-06	3.02%
STDS	50	500	1000	2000	5000		50	500	1000	2000	5000		Average	Standard Deviation	% RSD
7	0.00059	0.0068	0.01388	0.02783	0.06628		1.18E-05	1.36E-05	1.39E-05	1.39E-05	1.33E-05		1.32902E-05	8.74082E-07	6.58%
STDS	1000	2500	5000	10000	15000		1000	2500	5000	10000	15000		Average	Standard Deviation	% RSD
8	0.003584	0.008852	0.018227	0.035949	0.055657		3.58E-06	3.54E-06	3.65E-06	3.59E-06	3.71E-06		3.61511E-06	6.50166E-08	1.80%
STDS	100	250	1000	2500	5000		100	250	1000	2500	5000		Average	Standard Deviation	% RSD
9	0.02058	0.057636	0.220735	0.58131	1.189081		0.000206	0.000231	0.000221	0.000233	0.000238		0.000225484	1.2622E-05	5.60%

Table K 2: 4 Inch Membrane Calibration Verification			
All Laboratories			
RSD (pooled)	RSD Cal Max	k verification	k (ver) RSD (pooled)
4.45%	7.12%	2.2	16%

Table L: 10 Inch Membrane Calibration Verification																	
Lab	Standards Response					Calibration Factor (CF)					Single Laboratory			All Laboratories			
STDS	8	40	100	200	400	8	40	100	200	400	Average	Standard Deviation	% RSD	RSD (pooled)	RSD Cal Max	k verification	k (ver) RSD (pooled)
10	22.08433	120.8571	309.8668	653.16	1215.643	2.760541	3.021427	3.098668	3.2658	3.039108	3.037109	0.182228548	6.00%	9.00%	16.20%	2.4	39%
STDS	20	50	100	200	500	20	50	100	200	500	Average	Standard Deviation	% RSD				
6	0.057592	0.130322	0.265185	0.546313	1.428785	0.00288	0.002606	0.002663	0.002725	0.002858	0.002746	0.00011958	4.35%				
STDS	25	50	100	200	250	25	50	100	200	250	Average	Standard Deviation	% RSD				
9	0.110323	0.163399	0.33528	0.655487	0.86072	0.004413	0.003268	0.003353	0.003277	0.003443	0.003551	0.000487036	13.72%				

Table M 1: 4 Inch Membrane Initial and Ongoing Precision and Recovery																
LFB recovery								LFB Percent Recovery				IPR-OPR Calculation				
Lab	IDC Reading 1	IDC Reading 2	IDC Reading 3	IDC Reading 4	ODC Reading 1	Individual Laboratory Mean	Individual Laboratory Standard Deviation	Reading 1	Reading 2	Reading 3	Reading 4	ODC Reading 1	Individual Laboratory Mean	Individual Laboratory Standard Deviation	Individual Laboratory IPR Lower Limit	Individual Laboratory IPR Upper Limit
1	985.75	1018.92	1025.69	1029.08	852.95	982.48	74.42320115	98.58%	101.89%	102.57%	102.91%	85.30%	98.25%	7.44%	58.80%	137.69%
2	933.71	865.26	839.73	990.04	814.57	888.66	72.00515175	93.37%	86.53%	83.97%	99.00%	81.46%	88.87%	7.20%	50.70%	127.03%
3	973.9434	1041.9011	1057.4688	952.6322	933.53	991.90	54.9335672	97.39%	104.19%	105.75%	95.26%	93.35%	99.19%	5.49%	70.07%	128.30%
4	1053	1062	1046	1046	1040.00	1049.40	8.414273587	105.30%	106.20%	104.60%	104.60%	104.00%	104.94%	0.84%	100.48%	109.40%
5	990.556	973.416	956.671	974.063	942.77	967.50	18.2913463	99.06%	97.34%	95.67%	97.41%	94.28%	96.75%	1.83%	87.06%	106.44%
6	1022.209158	1069.326634	990.9106709	987.0689604	1016.58	1017.22	32.94564572	102.22%	106.93%	99.09%	98.71%	101.66%	101.72%	3.29%	84.26%	119.18%
7	914.38678	904.68693	899.01413	884.62246	954.38	911.42	26.32204961	91.44%	90.47%	89.90%	88.46%	95.44%	91.14%	2.63%	77.19%	105.09%
8	951.918	977.214	1021.866	947.834	921.95	964.16	37.74385319	95.19%	97.72%	102.19%	94.78%	92.20%	96.42%	3.77%	76.41%	116.42%
9	911.856219	909.634498	892.976666	889.473373	879.27	896.64	13.84618297	91.19%	90.96%	89.30%	88.95%	87.93%	89.66%	1.38%	82.33%	97.00%

Table M 2: 4 Inch Membrane Initial and Ongoing Precision and Recovery									
All Laboratory Mean	Pooled Within Laboratory Standard Deviation (sw)	Between Laboratory Standard Deviation of the Mean results (sb)	RSD IPR(max)	IPR Combined Standard Deviation (sc)	All Laboratory IPR Lower Limit	All Laboratory IPR Upper Limit	OPR Combined Standard Deviation (sc)	All Laboratory OPR Lower Limit	All Laboratory OPR Upper Limit
96.33%	4.41%	5.50%	7.79%	5.88%	82.79%	109.86%	7.02%	81.59%	111.06%

Table N 1: 10 Inch Membrane Initial and Ongoing Precision and Recovery																
LFB recovery								LFB Percent Recovery				IPR-OPR Calculation				
Lab	IDC Reading 1	IDC Reading 2	IDC Reading 3	IDC Reading 4	ODC Reading 1	Individual Laboratory Mean	Individual Laboratory Standard Deviation	Reading 1	Reading 2	Reading 3	Reading 4	ODC Reading 1	Individual Laboratory Mean	Individual Laboratory Standard Deviation	Individual Laboratory IDC Lower Limit	Individual Laboratory IDC Upper Limit
10	48.48	48.05	46.91	46.79	47.78	47.60	0.73	96.96%	96.10%	93.81%	93.59%	95.56%	95.21%	1.46%	87.44%	102.97%
6	51.33	48.78	46.65	47.49	48.84	48.62	1.77	102.66%	97.55%	93.30%	94.97%	97.68%	97.23%	3.55%	78.44%	116.02%
9	51.38	49.48	46.51	47.76	51.17	49.26	2.12	102.76%	98.97%	93.03%	95.51%	102.34%	98.52%	4.24%	76.02%	121.02%

Table N 2: 10 Inch Membrane Initial and Ongoing Precision and Recovery									
All Laboratory Mean	Pooled Within Laboratory Standard Deviation (sw)	Between Laboratory Standard Deviation of the Mean results (sb)	RSD IPR(max)	IPR Combined Standard Deviation (sc)	All Laboratory IDC Lower Limit	All Laboratory IDC Upper Limit	OPR Combined Standard Deviation (sc)	All Laboratory OPR Lower Limit	All Laboratory OPR Upper Limit
96.99%	3.30%	1.67%	6.36%	2.07%	90.37%	103.60%	3.53%	87.81%	106.16%

Table O 1: CCV Low Standard Bias and Precision 4 Inch Membrane															
CCV Reading						Difference			Squared Difference			Bias and precision			
Lab	CCV Low Reading 1	CCV Low Reading 2	CCV Low Reading 3	Standard Value (PPB N)	N	CCV Low Reading 1	CCV Low Reading 2	CCV Low Reading 3	CCV Low Reading 1	CCV Low Reading 2	CCV Low Reading 3	Single Lab Bias	Single Lab Precision	Total Lab Bias	Total Lab Precision
1	538.83	498.04	486.29	500.00	3	38.83	-1.96	-13.71	1507.77	3.84	187.96	8	29	-30	70
2	997.41	992.91	986.34	1000.00	3	-2.59	-7.09	-13.66	6.69	50.24	186.49	-8	11		
3	989.36	995.65	1001.16	1000.00	3	-10.64	-4.35	1.16	113.29	18.89	1.34	-5	8		
4	50.00	40.00	50.00	50.00	3	0.00	-10.00	0.00	0.00	100.00	0.00	-3	7		
5	507.53	505.67	506.16	500.00	3	7.53	5.67	6.16	56.73	32.19	37.92	6	8		
6	186.62	189.29	191.72	200.00	3	-13.38	-10.71	-8.28	179.12	114.64	68.60	-11	13		
7	468.15	454.21	469.00	500.00	3	-31.85	-45.79	-31.00	1014.11	2097.14	961.03	-36	45		
8	897.63	893.72	909.99	1000.00	3	-102.37	-106.28	-90.02	10480.23	11295.23	8102.70	-100	122		
9	2358.15	2332.34	2434.52	2500.00	3	-141.85	-167.66	-65.48	20120.93	28109.15	4287.61	-125	162		

Table O 2: CCV High Standard Bias and Precision 4 Inch Membrane															
CCV Reading						Difference			Squared Difference			Bias and precision			
Lab	CCV High Reading 1	CCV High Reading 2	CCV High Reading 3	Standard Value (PPB N)	N	CCV High Reading 1	CCV High Reading 2	CCV High Reading 3	CCV High Reading 1	CCV High Reading 2	CCV High Reading 3	Single Lab Bias	Single Lab Precision	Total Lab Bias	Total Lab Precision
1	1894.70	1936.85	1904.56	2000.00	3	-105.30	-63.15	-95.44	11088.09	3987.92	9108.79	-88	110	-55	136
2	1992.30	1984.49	1987.97	2000.00	3	-7.70	-15.51	-12.03	59.31	240.44	144.65	-12	15		
3	1966.67	2001.25	1932.49	2000.00	3	-33.34	1.25	-67.51	1111.22	1.57	4557.96	-33	53		
4	510.00	530.00	510.00	500.00	3	10.00	30.00	10.00	100.00	900.00	100.00	17	23		
5	1915.45	1921.10	1926.91	2000.00	3	-84.55	-78.91	-73.09	7148.70	6226.00	5342.44	-79	97		
6	501.14	486.86	495.04	500.00	3	1.14	-13.14	-4.96	1.30	172.69	24.62	-6	10		
7	947.53	859.03	874.55	1000.00	3	-52.47	-140.97	-125.45	2753.04	19873.54	15738.41	-106	139		
8	2560.87	2554.64	2539.59	2500.00	3	60.87	54.64	39.59	3704.67	2985.42	1567.37	52	64		
9	4961.73	4741.15	4591.29	5000.00	3	-38.27	-258.85	-408.71	1464.39	67000.83	167047.58	-235	343		

Table P: CCV Low and High Standard Bias and Precision 10 Inch Membrane																		
CCV							Difference				Squared Difference				Bias and Precision			
Lab	CCV Low Reading 1	CCV Low Reading 2	CCV Low Reading 3	CCV Low Reading 4	Standard Value (PPB N)	N	CCV Low Reading 1	CCV Low Reading 2	CCV Low Reading 3	CCV Low Reading 4	CCV Low Reading 1	CCV Low Reading 2	CCV Low Reading 3	CCV Low Reading 4	Single Lab Bias	Single Lab Precision	Total Lab Bias	Total Lab Precision
10	8.68	8.88	8.81	8.20	8.00	4	0.68	0.88	0.81	0.20	0.46	0.77	0.66	0.04	0.64	0.80	-2.28	4.90
6	17.95	18.87	18.18		20.00	3	-2.05	-1.13	-1.82		4.21	1.27	3.31		-1.67	2.10		
9	191.32	196.78	191.54		200.00	3	-8.68	-3.22	-8.46		75.28	10.35	71.61		-6.79	8.87		
CCV							Difference				Squared Difference				Bias and Precision			
Lab	CCV High Reading 1	CCV High Reading 2	CCV High Reading 3	CCV High Reading 4	Standard Value (PPB N)	N	CCV High Reading 1	CCV High Reading 2	CCV High Reading 3	CCV High Reading 4	CCV High Reading 1	CCV High Reading 2	CCV High Reading 3	CCV High Reading 4	Single Lab Bias	Single Lab Precision	Total Lab Bias	Total Lab Precision
10	38.80	38.49	37.86	38.28	40.00	4	-1.20	-1.51	-2.14	-1.72	1.45	2.28	4.58	2.96	-1.64	1.94	-0.10	10.00
6	45.64	48.25	46.37		50.00	3	-4.36	-1.75	-3.63		19.00	3.06	13.19		-3.25	4.20		
9	272.92	253.23	239.15		250.00	3	22.92	3.23	-10.85		525.19	10.44	117.62		5.10	18.07		

Table Q: Youden Pair Recovery-4 Inch Membrane						
Lab	**Youden A (Experimental Value)**	**Youden B (Experimental Value)**	**Youden A (Actual Value)**	**Youden B (Actual Value)**	**Youden A (Percent Recovery)**	**Youden B (Percent Recovery)**
4	2.51	9.02	2.45	8.80	102.45%	102.46%
8	9.67	2.68	8.80	2.45	109.91%	109.30%
1	0.82	3.88	0.68	3.68	121.02%	105.54%
2	0.62	8.48	0.68	8.80	90.92%	96.31%
3	3.85	2.66	3.68	2.45	104.64%	108.66%
5	3.72	0.67	3.68	0.68	101.15%	98.68%
8	0.73	2.51	0.68	2.45	107.11%	102.43%
9	3.34	2.25	3.68	2.45	90.65%	91.67%
6	10.54	1.05	10.66	1.10	98.84%	95.46%

Table R: Single Operator Standard Deviation and %RSD – 4 Inch Membrane			
M	2	2	2
2(M-1)	2	2	2
Difference (Di)	6.51	3.0609	1.19
	6.99	3.0511	1.09
Mean (D)	6.75	3.0560	1.14
Di-D	0.05933625	2.40002 E-05	0.002434069
	0.05933625	2.40002 E-05	0.002434069
Single Operator So (Standard Deviation)	0.24	0.0049	0.049336281
Single Analyst %RSD	4.08%	0.22%	1.63%

Table S:Youden Pair Recovery-10 Inch Membrane						
Lab	**Youden A (Experimental Value)**	**Youden B (Experimental Value)**	**Youden A (Actual Value)**	**Youden B (Actual Value)**	**Youden A (Percent Recovery)**	**Youden B (Percent Recovery)**
9	88.39	164.27	86.10	162.10	102.66%	101.34%
10	156.04	80.29	162.10	86.10	96.26%	93.25%
6	112.17	53.40	105.37	52.69	106.45%	101.35%

Table T: Single Operator Standard Deviation and %RSD – 10 Inch Membrane	
M	2
2(M-1)	2
Difference (Di)	75.89
	75.75
Mean (D)	75.82
Di-D	0.004657716
	0.004657716
Single Operator So (Standard Deviation)	0.07
Single Analyst %RSD	0.06%

Table U: Laboratory Reagent Blank – Instrument Detection Limit 4 Inch Membrane																							
Laboratory Reagent Blank Data									Difference			Squared Difference			Bias and Precision				Instrument Detection Limit					
Lab	Standard or Sample	Sample Concentration	Measurement 1	Measurement 2	Measurement 3	Average	Standard Deviation	N	Blank Reading 1	Blank Reading 2	Blank Reading 3	Blank Reading 1	Blank Reading 2	Blank Reading 3	Single Lab Bias	Single Lab Precision	Total Lab Bias	Total Lab Precision	Square Blank Reading 1 Difference from Mean	Square Blank Reading 2 Difference from Mean	Square Blank Reading 3 Difference from Mean	Total Lab Average	Pooled Standard Deviation	Instrument Detection Limits
1	Blank	0	3.2	2.2	3.4	3.0	0.6	3	3.17	2.24	3.45	10.07	5.00	11.87	2.95	3.67	7	19	0.05	0.51	0.24	7.3	14.7	44.0
2	Blank	0	-31.9	36.6	46.2	17.0	42.6	3	-31.93	36.63	46.24	1019.54	1341.82	2138.16	16.98	47.43			2392.24	386.14	856.15			
3	Blank	0	11.6	2.8	14.9	9.8	6.2	3	11.61	2.84	14.92	134.68	8.07	222.67	9.79	13.52			3.30	48.28	26.34			
4	Blank	0	4.5	13.0	-2.7	5.0	7.8	3	4.55	12.99	-2.69	20.66	168.85	7.21	4.95	9.92			0.16	64.68	58.32			
6	Blank	0	-0.2	1.1	3.4	1.4	1.8	3	-0.17	1.12	3.38	0.03	1.25	11.40	1.44	2.52			2.59	0.10	3.74			
6	Blank	0	4.1	5.6	3.0	4.2	1.3	3	4.06	5.55	3.00	16.51	30.81	9.01	4.21	5.31			0.02	1.81	1.45			
7	Blank	0	5.5	6.5	6.8	6.3	0.7	3	5.47	6.52	6.81	29.92	42.56	46.44	6.27	7.71			0.64	0.06	0.30			
8	Blank	0	-1.3	3.7	-1.3	0.4	2.9	3	-1.26	3.73	-1.26	1.59	13.89	1.59	0.40	2.92			2.77	11.06	2.77			
9	Blank	0	21.1	16.7	18.3	18.7	2.2	3	21.14	16.71	18.25	446.98	279.23	333.18	18.70	23.02			5.95	3.97	0.20			

Table V: Laboratory Reagent Blank – Instrument Detection Limit 10 Inch Membrane																							
Laboratory Reagent Blank Data									Difference			Squared Difference			Bias and Precision				Instrument Detection Limit					
Lab	Standard or Sample	Sample Concentration (PPB N as Ammonia)	Measurement 1	Measurement 2	Measurement 3	Average	Standard Deviation	N	Blank Reading 1	Blank Reading 2	Blank Reading 3	Blank Reading 1	Blank Reading 2	Blank Reading 3	Single Lab Bias	Single Lab Precision	Total Lab Bias	Total Lab Precision	Square Blank Reading 1 Difference from Mean	Square Blank Reading 2 Difference from Mean	Square Blank Reading 3 Difference from Mean	Total Lab Average	Pooled Standard Deviation	Instrument Detection Limits
10	Blank	0	-0.5	0.4	0.4	0.1	0.5	3	-0.49	0.40	0.44	0.24	0.16	0.20	0.12	0.55	-0.05	0.37	0.37	0.08	0.10	-0.05	0.31	0.9
6	Blank	0	-0.3	-0.1	-0.3	-0.3	0.1	3	-0.34	-0.10	-0.34	0.12	0.01	0.12	-0.26	0.35			0.01	0.03	0.01			
8	Blank	0	0.0	0.0	0.0	0.0	0.0	3	0.00	0.01	0.01	0.00	0.00	0.00	0.01	0.01			0.00	0.00	0.00			