

SOP-C-103

**Determination of Total Kjeldahl
Nitrogen and Total Phosphorus**

Revision 16

Approval:



Laboratory Manager

3-26-24

Date



Concurrence

3/26/2024

Date

Effective date: 3-26-24

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Texas Institute for Applied Environmental Research

Determination of Total Kjeldahl Nitrogen and Total Phosphorus

i. Identification of the method

- a. Total Kjeldahl Nitrogen and Total Phosphorus by SM 4500N_{org}B, 4500NH₃G (approved 2018); EPA 365.4 (approved 1974), 365.3 (approved 1978)

ii. Applicable matrix or matrices

- a. nonpotable water and wastewater samples, or aqueous solutions and suspensions, including solid extracts
- b. TNI accreditation only applies to the nonpotable water samples. Data produced on other matrices are nonaccredited.

iii. Limits of detection and quantitation

- a. Limits of ranges vary and are bracketed by the calibration standards. LODs are determined annually.

iv. Scope and application, including parameters to be analyzed

- a. This procedure applies to water and wastewater samples, filters of samples, extracts of soil, sediment, sludge or other solids received for laboratory analysis

v. Summary of the method

- a. Spectrophotometric autoanalysis of a catalytically digested sample. Organic forms of N & P are broken down into inorganic forms for colorimetric determination.
- b. Copper is used in place of mercury as the catalyst called for in some older methods.

vi. Definitions

- a. Carrier solution: used to wash out the sample lines between samples to prevent carryover.
- b. Flow Injection (FIA): Introduction of sample into reagents stream for analysis. Method of analysis for the QuikChem 8000 Analyzer.
- c. Deionized water (DI): water passed through ion exchange resin that meets Type II standards (specific conductance <1.0 µS/cm).
- d. Cycle time: The wash time plus the sample time. Also, the Sample load time plus Inject time on the manifold injection valve.

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- e. Standard QA/QC definitions are found in QAM-Q-101, "Laboratory Quality Control".
- f. DOP: Demonstration of Performance

vii. Interferences

- a. Nitrate levels in excess of 10 mg/L, very high quantities of inorganic salts or solids
- b. Samples with high or complex organic content may need extended digestion times to avoid boil over.
- c. Sample color with an absorbance in the range of the color that develops for analysis.

viii. Safety

- a. All aspects of this procedure comply with QAM-S-101, "Laboratory Safety".
- b. Rubber gloves, safety glasses, and a labcoat should be worn when handling samples and reagents.
- c. Sodium nitroferricyanide - protective equipment: A dustmask (or equivalent), rubber gloves, protective clothing and appropriate ventilation is used as needed. Wash gloves thoroughly before removing.
- d. Sulfuric acid - protective equipment: Face shield is required for working with concentrated acids; labcoat and rubber gloves are also worn as needed.
- e. Sodium hydroxide- extremely caustic; gloves and face shield are recommended.

ix. Equipment and supplies

- a. See QAM-I-102, "The Operation and Calibration of the Autoanalyzer".
- b. See QAM-I-107, "The Operation and Calibration of the Block Digester".
- c. Class A glassware is used in standard and reagent preparations.
- d. Filter paper (Whatman 41 or equivalent)
- e. Standard general labware- class A for volume measurement of sample
- f. Water bath (solid digestion)
- g. Hot plate (solid digestion)

x. Reagents and standards

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- a. Sulfuric acid (20%V:V) (500 mL)- Add 100 mL of concentrated sulfuric acid to 350 mL of DI and dilute to 500 mL in a volumetric flask. Cool to room temperature before using.
- b. Copper sulfate (500 mL) - Dissolve 45 gm of cupric sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in 350 mL of 20% sulfuric acid and dilute to 500 mL in a volumetric flask using the 20% sulfuric acid solution. Mix well.
- c. Block Digestion Solution (2L) for TKN/TP
 1. Add 400 mL of conc. sulfuric acid to 1200 mL of DI.
 2. Add 266 gm of potassium sulfate (K_2SO_4) to the acid solution and stir to dissolve.
 3. Add 50 mL of the copper sulfate solution and mix well.
 4. Cool the mixture to room temperature, transfer the mixture to a 2-Liter volumetric flask, dilute to mark with DI and mix well.
- d. Autoanalyzer Reagents for Total Kjeldahl Nitrogen
 1. 0.8 N Sodium hydroxide (NaOH) - Add 32-g of nitrogen-free NaOH to 800 mL of deionized water in a 1-L volumetric flask. Dilute to 1-L when the NaOH has dissolved using DI. Store in a tightly capped plastic container.
 2. Stock potassium sodium tartrate solution ($\text{KNaC}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$) (1 L) - In a 1-Liter volumetric flask dissolve 200 g of potassium sodium tartrate in 800 mL of DI. Dilute to 1 L with DI and mix well.
 3. Stock buffer, sodium phosphate dibasic (Na_2HPO_4) (1 L) - Add 20 g of NaOH to about 800 mL of DI in a 1-L volumetric flask. Dissolve 134 g of sodium phosphate dibasic (anhydrous) or 253 g of Sodium Phosphate dibasic heptahydrate ($\text{Na}_2\text{HPO}_4 \cdot 7\text{H}_2\text{O}$) in the solution. After all has dissolved, dilute to 1 Liter with DI and mix well.
 4. Working buffer (1 L) for TKN
 - i. Add 200 mL of stock buffer to a 1-L volumetric flask.
 - ii. Add 250 mL of stock potassium sodium tartrate to the flask while stirring the solution.
 - iii. Add 32 g of NaOH pellets slowly while stirring the solution.

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- iv. Dilute the solution to 1 Liter with DI and filter through a 0.45-micron filter if necessary.
- v. Prepare the Working Buffer Solution fresh daily.
- 5. Salicylate ($\text{NaC}_7\text{H}_5\text{O}_3$)/nitroferricyanide solution ($\text{Na}_2\text{Fe}(\text{CN})_5$):
 - i. Dissolve 150 g of sodium salicylate and 1.0 g of sodium nitroferricyanide dihydrate in 300 mL of DI.
 - ii. Filter the solution through fast filter paper (Whatman 41 or equivalent) into a 1-L volumetric flask.
 - iii. Dilute to the mark with DI and mix well. Store the solution in an amber bottle.
- 6. Sodium hypochlorite solution:
 - i. Add 25 mL of sodium hypochlorite (6% NaOCl , example is Clorox™) to a 500 mL volumetric flask. Alternatively use 28 mL of 5.25% bleach or 20 mL of 7.5% bleach.
 - ii. Dilute to volume with DI and mix well. Transfer to an amber glass bottle. Prepare fresh daily.
- 7. Carrier solution (2 L):
 - i. Add 100 mL of concentrated sulfuric acid to 1,600 mL of DI in a 2-Liter volumetric flask, while stirring.
 - ii. Add 4 mL of Copper Sulfate solution.
 - iii. Cool the solution, dilute to 2 L with DI and mix well.
- e. Autoanalyzer Reagents for Total Phosphorous
 - 1. Sodium chloride /sulfuric acid solution:
 - i. Add 50 mL of concentrated sulfuric acid to 800 mL of DI water in a 1-L volumetric flask, while stirring.
 - ii. Add 30 gm of Sodium Chloride (NaCl). Cool the solution, dilute to 1 liter with DI and mix well.
 - 2. Molybdate/antimony solution (1 L): Add 8 gm of ammonium molybdate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot \text{H}_2\text{O}$) and 0.2 gm of antimony potassium tartrate to 800 mL of DI. Dilute to 1 Liter with DI.
 - 3. Ascorbic acid solution (1 L):
 - i. Add 60 g of ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$) to 800 mL of DI in a 1 L volumetric.
 - ii. Add 2 mL of Acetone ($\text{C}_3\text{H}_6\text{O}$) and dilute to 1 L with DI. Store in an amber bottle in the refrigerator.

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4. Carrier – Use DI water as the carrier for the Total Phosphate analysis.
- f. TP digestion of solids
 1. 11N Sulfuric acid: Slowly add 310 mL conc. H_2SO_4 to approximately 600 mL DI. Cool and dilute to 1 L.
 2. 1.0 N H_2SO_4 : Slowly add 3 mL of concentrated H_2SO_4 to about 80 mL DI water. Cool and dilute to 100 mL.
 3. Sodium bisulfite solution: Dissolve 5.2 g of NaHSO_3 in 100 mL of 1.0 N H_2SO_4 .
 4. Ammonium persulfate
- g. Ammonia nitrogen /Potassium dihydrogen phosphate stock solution 1000 mg/L (N-P) (1 L):
 1. Dry approximately 6 g of ammonium chloride (NH_4Cl) and 6 g of potassium dihydrogen phosphate (KH_2PO_4) at 104°C for 2 hr. Cool and store in a desiccator.
 2. Add 3.819 g of dried ammonium chloride and 4.393 g of dried potassium dihydrogen phosphate to 900 mL of DI and dilute to 1 L in a volumetric flask with DI.
 3. Preserve with 2 mL of chloroform (CHCl_3) and refrigerate at $>0^\circ\text{C}$ to $\leq 6^\circ\text{C}$. Solution is good for 6 months.
- h. Ammonia nitrogen/potassium dihydrogen phosphate working solution 100 mg/L (WS N-P)(1 L):
 1. Dilute 50 mL of N-P stock to 500 mL in a volumetric flask using DI.
 2. Add 0.5 mL of concentrated sulfuric acid as a preservative.
 3. Label the working solution as follows: Contents (ex. "N-P working solution"), "mmddy" (Where mmddy is the date stamp the solution was prepared, "a" is the sequence value of the standard prepared that day. Second will be "b", 26th will be "z", 27th will be "aa", etc.), analyst initials, and concentration in mg/L.
- i. Prepare calibration standards from the 100-mg/L N-P working solution. All standards are preserved to pH<2 with up to 1% by volume concentrated Sulfuric Acid.
- j. Make the following standards for the TKN/TP analysis: 0.06 (TP LOQ), 0.2 (TKN LOQ), 0.5, 0.8, 1.0, 2.0, 5.0, 8.0 and 10.0 mg/L. Standards are digested before use.

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<i>Standards (mg/L)</i>	<i>mL made</i>	<i>Amount WS used</i>
0.06 (TP only)	250	0.15 mL
0.2	250	0.5 mL
0.5	250	1.25 mL
0.8	250	2.0 mL
1.0	250	2.5 mL
2.0	250	5.0 mL
5.0	250	12.5 mL
8.0	250	20.0 mL
10.0	250	25.0 mL
5.0 ICV/CCV	1000	5 mL CCV stock
5.0 TP LCS	1000	5 mL TP LCS stock
5.0 TP LCSD	1000	5 mL TP LCS Stock
5.0 TKN LCS	1000	5mL TKN LCS Stock
5.0 TKN LCSD	1000	5mL TKN LCS Stock

- k. Continuing Calibration Stock Solutions for TP and TKN (1000 mg/L) (CCV) - Prepare as specified by the laboratory manager from a source other than that of the calibration standards, or purchase commercially.
- l. Prepare a 5.0 mg/L ICV/CCV solution by diluting 5.0 mL of the CCV stock. Make up to 1000 mL using DI in a volumetric flask after adding up to 1% by volume concentrated sulfuric acid for preservation.
- m. Laboratory Control Standard Stock Solution for TP (1000 mg/L):
 1. Dry approximately 5 g of Adenosine Monophosphate (AMP) at 104°C for 2 hr. Cool and store in a desiccator.
 2. Add 3.351 g of dried AMP to 200 mL of DI and dilute to 250 mL in a volumetric flask with DI.
- n. Prepare a 5.0 mg/L TP LCS by diluting 5.0 mL of the TP LCS stock, to 1000 mL using DI in a volumetric flask after adding up to 1% by volume concentrated sulfuric acid for preservation. A duplicate of the LCS (known as TP LCSD) is also used.
- o. Laboratory Control Standard Stock Solution for TKN (1000 mg/L):

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1. Dry about 3 g of L-Lysine dihydrochloride (LYS) at 104°C for 2 hr. Cool and store in a desiccator.
2. Add 1.955 g of dried LYS to 200 mL of DI and dilute to 250 mL in a volumetric flask with DI.
- p. Prepare a 5.0 mg/L TKN LCS by diluting 5.0 mL of the TKN LCS stock to 1000 mL using DI in a volumetric flask after adding up to 1% by volume concentrated sulfuric acid for preservation. A duplicate of the LCS (known as TKN LCSD) is also used.
- q. Prepare a 0.06 mg/L TP LOQ by diluting 0.06 mL of the TP LCS stock to 1000 mL using DI in a volumetric flask. Add up to 1% by volume concentrated sulfuric acid to the standard for preservation.
- r. Prepare a 0.2 mg/L TKN LOQ by diluting 0.2 mL of the TKN LCS stock to 1000 mL using DI in a volumetric flask. Add up to 1% by volume concentrated sulfuric acid for preservation.
- s. For Low Range TP make 500 mL each of 0.01, 0.02, 0.05, 0.10, 0.20, and 0.50 mg/L standard. Prepare a 0.2 mg/L each of ICV/CCV, LCS and LCSD, and a 0.01 mg/L LOQ. Acidify dropwise until the pH<2 (about 6 drops per 500 mL).

Standard (mg/L)	mL made	Amount WS used
0.01	500	0.05
0.02	500	0.10
0.05	500	0.25
0.10	500	0.50
0.20	500	1.0
0.50	500	2.5
0.20 ICV/CCV	500	0.10 CCV stock
0.20 LCS/LCSD	500	1.0 Low TP LCS stock
0.01 LOQ	500	0.05 Low TP LCS stock

- t. Low TP LCS Stock: dilute 10 mL of the TP LCS stock to 100 mL using DI in a class A volumetric flask after adding up to 1% by volume concentrated sulfuric acid for preservation.

xi. Sample collection, preservation, shipment and storage

- a. Holding Time: 28 days from collection-water, 6 months-soil

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- b. Preservation: Refrigerate sample to $>0-6^{\circ}\text{C}$; $\text{pH} < 2$
 H_2SO_4 for water, none for soil
- c. Samples are not collected or normally transported by the TIAER laboratory.

xii. Quality control

- a. All data are documented and maintained in accordance with QAM-A-102, "Document and Data Control".
- b. If any spike, duplicate, or standard does not pass the acceptance criteria as described in QAM-Q-101, "Laboratory Quality Control", reanalyze if possible or complete a Corrective Action Report in accordance with QAM-Q-105, "Corrective Actions".
- c. The standards are valid for twenty-eight (28) days after preparation. Each calibration standard used in the curve is 75-125% of the linear value. The LOQ is also 75-125% of the true value. If used, LOQ calibration verification is 70-130% of the true value.
- d. Record the calibration standards in the Standards Logbook. Record reagents in the Reagents Logbook.
- e. The standard curve has a minimum of 4 reference points. The range of the standards for the TKN and TP test is between 0.06 to 10.0 mg/L.
 - Low range TP: 0.01-0.5 mg/L
 - High Range TP: 0.06-10.0 mg/L
 - Normal Range TKN: 0.2 – 10.0 mg/LRange may be extended lower with concentration or higher with dilution of samples and standards. Other concentrations may be used as required if approved by the Lab Manager.
- f. The baseline is within \pm the set LOQ limit for the analysis.
- g. For TKN analysis analyze an aliquot of the carrier after the ICB to ensure that the carrier isn't causing errors with sample dilutions. If the carrier concentration is within \pm the set LOQ limit proceed with analysis.
- h. All aspects of this procedure comply with QAM-Q-101, "Laboratory Quality Control".
- i. Quality control calculations are performed using the appropriate quality control portion (TKN or TP) located in the current LIMS.

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- j. Refer to the appropriate instrument manual for specific manifold and flow diagrams.
- k. Procedure modifications for mercury from EPA 365.4 & EPA 351.2 are no longer used as the SM method is now employed. Samples are not distilled per SM 4500 NH₃G. Samples are shaken in the bottle rather than exposing to possible contamination by ammonia in a vortex mixer. Boiling chips are Hengar™ specialized and not Teflon™, which tend to float at the top of the sample during digestion. All references to SFA instrumentation have been removed in this revision.
- l. For TP digestion of solids, modifications include analyzing the digestate using standards in the range of 0.06-10.0 mg/L P rather than 0.01 to 1.2 (project requirements) and measuring the absorbance at 880 nm rather than 650 nm (see DOP reference).

xiii. Calibration and standardization

- a. Continue as described in the Procedure section below.
- b. Check that analysis is ready to proceed as detailed in QAM-I-102, "Operation and Calibration of the Autoanalyzers".
- c. Computer start-up – Follow directions in QAM-I-102, "Operation and Calibration of the Autoanalyzers" according to the instrument being used for the analysis.

xiv. Procedure

- a. Digestion Procedure
 - a. See the QAM-I-107, "Operation and Calibration of the Block Digester" for the operation and calibration of the block digester.
 - b. Preparation of digestion tubes for block digestion.
 - 1. Block 1 contains 2 calibration blanks, 9 standards, LOQ and a sample duplicate and spike/spike duplicate for every ten samples. Blocks 2 & 3 continue with samples and required QC. Method blanks, LOQ and LCS/LCSDs are included for each preparation batch (PB), but are not counted part of each preparation or run batch. The MB & LCS/LCSD designate the start of each sample PB of 20 samples or less. Field splits or field dups are included in the

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order received by the laboratory and are not part of the sample prep batch set of 20 or less actual environmental samples.

2. Measure 25 mL aliquots of each standard, sample, field split (if present), spike, or blank using a graduated cylinder and pour into digestion tubes. For solids, weigh wet aliquots of 5.0 grams, or aliquots prepared dry in accordance with SOP-C-131, "Preparation of Soil Samples", and then rinse into digestion tube with a minimal amount of DI water.
3. Prepare spikes by adding an appropriate amount of spiking solution to 25 mL of the sample or dilution of the sample according to the table:

<i>Desired Analyte Range</i>	<i>mL of Spike (spike mg/L)</i>	<i>Solution Concentration (mg/L)</i>
Low range Total Phosphorus	1.0 (0.2)	100
High range TP and TKN	1.0 (4.0)	100
Ultra high range TKN and TP and solid samples	1.0 (40)	1000

4. Add 5 mL of digestion solution to each tube.
 5. Add two or more boiling chips to each digestion tube to prevent "bumping" and loss of sample.
 6. Wash the tube walls with a few mL of DI before digestion, if needed. Swirl tubes to mix.
 7. Set the initial temperature at 160°C and digest for 3 hours. Another lower initial temperature stage may be required for more difficult matrices.
 8. Set the temperature at 380°C and digest for 3 hours.
 9. Set the final temperature to 98°C and allow tubes to cool to this temperature before proceeding.
- c. Post digestion (digestion may be automated)

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1. Add 5 to 10 mL of DI to the digestion tubes to prevent crystallization of the sample/reagent mixture and to redissolve it.
 2. Allow 60-90 minutes at 98°C after the DI addition for the sample/reagent mixture to dissolve.
 3. Pour each sample into a 25-mL graduated cylinder.
 4. Rinse each tube with three 5-6 mL portions of DI water, adding each rinse to the graduated cylinder.
 5. Bring each sample volume to 25 mL with DI.
 6. Pour each sample into the container labeled with its corresponding sample number.
 7. Place the samples into a plastic tub and refrigerate until analysis.
 8. Before analysis, turbid digestates are filtered to remove any fine particulates that may interfere with accuracy.
 9. Rinse the graduated cylinder at least three times with DI between each sample.
- d. Digestion of Low TP samples
1. Add aliquots of about 50 mL to each digestion tube and heat at 160°C until reduced. Continue adding aliquots and reducing for a total volume of 500 mL for each sample, standard and blank.
 2. Spike one sample per QC batch with 1.0 mL of WS for a 0.2 mg/L spike
 3. Perform digestion as described above.
- e. Digestion of Solids
1. Transfer 50 mL of sample or a weighed solid aliquot diluted into 50 mL into a 125 mL Erlenmeyer flask and add 1 mL of 11 N H₂SO₄.
 2. Add 0.4 g ammonium persulfate, mix and boil gently for about 30-40 minutes or until a final volume of about 10 mL is reached. Cool and dilute to about 40 mL and filter.
 3. For samples containing arsenic or high levels of iron, add 5 mL of sodium bisulfite solution, mix and place in a 95°C water bath for 30 minutes (20 minutes after the temperature of the sample reaches 95°C). Cool and dilute to 50 mL.

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b. Analysis Procedure

a. Instrument start-up

1. See QAM-I-102, "Operation and Calibration of the Autoanalyzers", for instrument operation and calibration.
2. Connect the reagent lines for each test to DI water for flushing prior to analysis.
3. Refer to the manufacturer's instrument manual for correct analysis module setup.
4. The LOQ is placed as the lowest calibration standard at the beginning of an analytical run once each day samples are analyzed (ARB) and passes acceptance criteria before continuing the run.

b. Introduction of Reagents

1. Change the reagent lines from the manifold solution or DI to the reagents themselves.
2. For the TKN analysis connect all the reagents except for the salicylate/nitroferricyanide solution.
3. For the TP analysis connect all the reagents except for the molybdate/antimony solution.
4. Turn the manifold heaters on and make sure they are set at the proper temperatures (37°C for TP, 60°C for TKN).
5. After the reagents have been in the system for fifteen minutes, check the pH from the waste lines after the detector. The pH of the TKN line is > 10, and the pH of the TP line is < 2.
6. If pH values are correct, begin feeding the salicylate/nitroferricyanide reagent for TKN analysis and molybdate/antimony solution for TP. If incorrect, check solution and reagent feed lines before remaking reagents. Correct any kinks, worn tubing or errors before proceeding.
7. Let the systems equilibrate to the reagents (Approximately 20-30 minutes).
8. Check that analysis is ready to proceed as detailed in QAM-I-102, "Operation and Calibration of the Autoanalyzers".
9. Computer start-up – Follow directions in QAM-I-102, "Operation and Calibration of the Autoanalyzers"

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according to the instrument being used for the
analysis.

c. Sample Table

1. Load analysis template and enter sample table and standard information as detailed in QAM-I-102.
2. Use digestion map to fill entries in the sample table
3. Save the table as directed in QAM-I-102 according to the instrument being employed.
4. Print the table as detailed in QAM-I-102 according to the instrument being employed.

d. Analysis of Samples

1. Follow directions for data collection as found in QAM-I-102.
2. Check ICV/CCV and other cup levels during analysis. Replenish sample cup if level becomes too low for adequate sampling.
3. Upon completion of the run, the message: "Tray Run Complete" is displayed at the bottom of the run file screen.

e. Flow solution shut down procedure.

1. Remove the reagent line from the molybdate/antimony solution and place into the TP manifold solution.
2. Remove the reagent line from the salicylate/nitroferricyanide and place into the DI.
3. After 5 minutes, move the carrier lines from the reagent to the wash solution. Allow the system to flow for about 5 minutes.
4. Remove the rest of the reagent lines and connect to the appropriate manifold or rinse solution.
5. Flush the system for about 35 minutes at analysis setting for pump.
6. Turn off the pump.
7. Place the line tensioners in the lowest position and release them from the platens on the pump.
8. Exit from the analysis program on the computer.
9. Turn off spectrophotometer unit at main switch.

f. Computer shut down

1. Close the Tray window. Answer "No" to questions asking to save changes to Method and Tray. At Data

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System window, click on LogOff. Click on "Yes" to the question. At the System Entry window, click on "Exit". Again, answer "Yes" to question. Click on "START" at the bottom of the screen and highlight "Shutdown".

xv. Data analysis and calculations;

- a. Follow instructions in QAM-I-102, "Operation and Calibration of the Autoanalyzers," to produce final report of analysis and to export results for entry into project data file for calculation by the LIMS.

xvi. Method performance

- a. refer to QAM-Q-101, "Laboratory Quality Control"

xvii. Pollution prevention

- a. refer to QAM-W-101, "Disposal of Laboratory Waste"

xviii. Data assessment and acceptance criteria for quality control measures

- a. refer to QAM-Q-101, "Laboratory Quality Control"

xix. Corrective actions for out-of-control data

- a. refer to QAM-Q-105, "Corrective Actions"

xx. Contingencies for handling out-of-control or unacceptable data

- a. refer to QAM-Q-105, "Corrective Actions"

xxi. Waste management

- a. refer to QAM-W-101, "Disposal of Laboratory Waste"

xxii. References

- a. "Flow Injection Analyzer Training Manual", QuikChem 8000 Automated Ion Analyzer Continuum Series, Zellweger Analytics, March, 1999.
- b. "Methods Manual", QuikChem Methods, Zellweger Analytics, October, 1997.
- c. Standard Methods for the Analysis of Water and Wastewater, latest *online edition*, 4500- $N_{org}B$ and 4500- NH_3G (approved 2018).

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- d. Method of Chemical Analysis of Water and Wastes, John R. Kopp, et al., Environmental Monitoring and Support Lab, Cincinnati, OH., 365.3 (1978) & 365.4 (1974).
- e. The National Environmental Laboratory Accreditation Conference Institute (NELAP) standard, 2016.
- f. Demonstration of Performance (DOP) for method modification of EPA 365.3 wavelength change, Logbook 10-002, p. 5.

xxiii. Any tables, diagrams, flowcharts and validation data

- a. FIA Parameters for TP and TKN

Working Copy

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Attachment 1
FIA Parameters for TP and TKN

TEST	TKN	TP
Low Range mg/L	NA	0.01-0.5
High Range mg/L	0.2-10.0	0.06-10.0
PUMP		
Pump speed	35 %	35 %
Pump tubes		
Buffer	yel/yel	orn/wh
Salicylate	wh/wh	
		Molybdate
Chlorox	blk/blk	orn/orn
Pull off	purple	purple
Sample	green	green
Carrier	orn/orn	red/red
NaOH	orn/orn	Ascorbic acid
		orn/orn
DETECTOR		
Wavelength	660nm	880nm
SAMPLER		
Rate	<u>45 samples/hr</u>	<u>45 samples/hr</u>
Sample time	20 sec	20 sec
Wash Time	45 sec	45 sec