DETERMINATION OF TOTAL KJELDAHL NITROGEN IN WATER USING THE SEAL AQ2 DISCRETE ANALYZER

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1. SCOPE AND APPLICATION

1.1. The Seal AQ2 Discrete Analyzer is used for the determination of total Kjeldahl Nitrogen (TKN) in ground, surface, and saline waters, and domestic and industrial wastes. The calibration range is from the method detection limit (MDL) 0.080 to 4.0 mg N/L. The range may be extended by dilution up to a limit of 10 mg N/L of organic N in the sample prior to digestion. The practical quantitation limit (PQL) is 0.20 mg N/L.

1.2. The tests covered by this SOP are W-TKN and W-TKN-F.

2. SUMMARY OF THE METHOD

2.1. This SOP is based on EPA method 351.2, Rev. 2.0 (1993) and Seal AQ2 method EPA-111-A Rev. 4.

2.2. Prior to analysis, digestion converts free ammonia and organic nitrogen compounds to ammonium sulfate (DEP SOP NU-091).

2.3. Ammonium reacts with salicylate and hypochlorite in a buffered, alkaline solution in the presence of sodium nitroferricyanide (pH = 12.4-12.7) to form the salicylic acid analog of indophenol blue. The blue-green color produced is measured at 660 nm.

2.4. Interferences: The working buffer eliminates

precipitation of calcium and magnesium hydroxides. Samples that are turbid must be filtered prior to determination. High nitrate concentrations (>10 mg N/L) interfere with the TKN preparation procedure. The interference can be either negative or positive. For any samples with a known nitrate concentration of 10 mg N/L or higher the TKN result should be qualified with a J qualifier or the sample diluted sufficiently to decrease the nitrate concentration to below 10 mg N/L PRIOR to digestion.

3. EQUIPMENT AND SUPPLIES

3.1. Instrumentation - Seal AQ2 Discrete Analyzer.

3.2. Glassware and pipettes - Class A type volumetric glassware and analytical pipettes.

Stock standard:	1 - 1 L volumetric flask
Working standards:	1 - 100 mL volumetric flask 4 - 250 mL volumetric flasks
Reagents:	3 - 500 mL Nalgene bottles with caps 2 - 500 mL volumetric flasks

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2 - 100 mL volumetric flasks
1 - 50 mL volumetric flask
2 - 50 mL graduated cylinders
2 - 1 L volumetric flasks
1 - 1 L beaker
2 - 1 L Nalgene bottles
1 - 500 mL brown Nalgene bottle

Misc.: magnetic stirring apparatus and stir bar
spatulas for weighing out reagents
variable Eppendorf type pipettes
vacuum filtration apparatus

4. REAGENTS AND STANDARDS (SEE DEP SOPS NU-041 and NU-042)

Note: Wash all glassware with 1:2 HCl and rinse at least three times with deionized (DI) water. Before preparing reagents or standards, or running samples, read the safety notes in the safety/hazardous waste section of this SOP and the MSDS for the chemicals used in this analysis.

Note: All reagent preparations must be documented in the Reagent and Standard Logsheet. This excel sheet is located in the TKN-Cu folder in the REAGENT_PREP folder on the Nutrients drive, and needs to be updated whenever a reagent is newly prepared. All reagent and standard preparations must be documented in the LIMS 'Standard Preparation Tracker' module. Unique ID numbers for each standard and reagent are generated in the module and documented in the prep worksheet in LIMS or in the Reagent and Standard Logsheet submitted with each analysis folder. If available, a Certificate of Analysis for each purchased standard or reagent is also maintained in the REAGENT_PREP folder. A Certificate of Analysis for the standard reference material (4.3.4.) is kept in a binder in C307. All containers holding standards or reagents must be labeled with their unique ID and expiration date.

Note: The volumes prepared for the reagents and standards may be altered by the analyst as necessary. The final concentrations of the constituents of a standard or reagent must not be changed.

4.1. Chemicals:

4.1.1. Ammonium Chloride, NH_4Cl

4.1.2. DI Water, ammonia-free

4.1.3. Glycine, $C_2H_5NO_2$

4.1.4. Sodium Salicylate, $NaC_7H_5O_3$

4.1.5. Sulfuric Acid, concentrated, H_2SO_4

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4.1.6. Sodium Hydroxide, NaOH

4.1.7. Sodium Hypochlorite (Clorox), NaOCl (approx. 8.25 % NaOCl)

4.1.8. Sodium Nitroferricyanide (Nitroprusside), $Na_2Fe(CN)_5NO*2H_2O$

4.1.9. Sodium Chloride, NaCl

4.1.10. Sodium Potassium Tartrate, $KOCO(CHOH)_2COONa*4H_2O$

4.1.11. Sodium Phosphate, Dibasic Heptahydrate, $Na_2HPO_4*7H_2O$

4.1.12. β-Glycerophosphoric Acid, Disodium Salt, Pentahydrate, $C_3H_7Na_2O_6P*5H_2O$

4.1.13. Potassium Dihydrogen Phosphate, KH_2PO_4

4.1.14. Standard Reference Material (ALTQC)

4.2. Reagents:

4.2.1. **Stock sodium hydroxide, 20 % solution** - In a 1 L beaker, add 200 g NaOH (4.1.6.) to approx. 800 mL DI water while stirring slowly. Bring to volume with DI water in a 1 L volumetric flask. Do not filter. Holding time, 180 days.

Note: Solution will become hot as sodium hydroxide is added to water. Use a plastic tray containing water as water bath to cool the NaOH solution.

4.2.2. **Stock buffer** - Using a 1 L beaker and magnetic stir plate, add 254.0 g sodium phosphate dibasic heptahydrate (4.1.11.) and 20 g NaOH (4.1.6.) to approx. 700 mL DI water. Stir until dissolved, approx. 10 min. Transfer to a 1 L volumetric flask and bring to volume with DI water. Store at room temperature (if the reagent is stored cold, precipitation can occur). Holding time, 60 days.

4.2.3. **Sodium potassium tartrate, 10 % solution** - In a 2 L beaker, dissolve 200 g of sodium potassium tartrate (4.1.10.) in approx. 1.2 L of DI water. Add two to three pellets of NaOH (4.1.6.) and a few boiling chips, boil gently for 45 minutes to drive away any residual ammonia contamination. Cover, and after the solution has cooled down to room temperature, bring it to volume in a 2 L volumetric flask. Adjust the pH to that of DI water (approx. pH 6-7) by adding conc. H_2SO_4 (4.1.5.) dropwise, until the desired pH is reached. Holding time, 180 days.

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4.2.4. **Working/stock salicylate/nitroferricyanide** - Use caution when weighing out sodium salicylate; it may have a small particle size and be dusty.

4.2.4.1. **Stock color reagent (sodium nitroferricyanide solution; 30 g/L)** - Dissolve 1.5 g sodium nitroferricyanide (4.1.8.) in a 50 mL volumetric flask and bring to volume with ammonia free DI water. Store the solution in a brown bottle in a refrigerator. Holding time, 30 days. Reprepare if the solution develops a blue-green tint.

4.2.4.2. **Stock alkaline sodium salicylate solution** - Using a 1 L beaker and magnetic stirring apparatus, dissolve 75 g anhydrous sodium salicylate (4.1.4.) and 10 g sodium hydroxide (4.1.6.) in approx. 350 mL DI water. The mixture will take approx. 20 minutes to dissolve. Transfer to a 500 mL volumetric flask and bring to volume. Store at room temperature in a brown bottle. Holding time, 60 days.

4.2.4.3. **Working color reagent (salicylate/nitroferricyanide solution)** - To 40 mL of the stock sodium salicylate solution (4.2.4.2.), add 0.25 mL of the stock sodium nitroferricyanide solution (4.2.4.1.). This working reagent may be prepared/mixed directly in a 40 mL (wedge) AQ2 reagent reservoir. Holding time, 2 days.

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4.2.5. **Working buffer** - Use a clean and empty 100 mL volumetric flask and combine the reagents in the stated order. Add 50 mL of 10 % stock sodium potassium tartrate (4.2.3.) to 20 mL of stock buffer solution (4.2.2.) and mix. Add 18.4 mL of 20 % NaOH (4.2.1.) solution to the mixture. Dilute to the mark with DI water and mix well. Holding time, 3 days.

4.2.6. **Working hypochlorite** - To a 100 mL volumetric flask containing approx. 50 mL DI water, add 14.54 mL hypochlorite (4.1.7.). Bring to volume. Please check the hypochlorite concentration of the Clorox used and adjust the volume used as necessary. Holding time, 3 days.

4.3. Standard and spiking solutions:

4.3.1. **NH_3 stock (1000 mg N/L) for working standards** - To a 1 L volumetric flask containing about 500 mL DI water, add 3.819 g ammonium chloride (4.1.1.), which has been dried for 12-24 hours at 105 ^oC. Dilute to the mark and invert 12 times. Refrigerate. This solution is also used for ammonia analysis (DEP SOP NU-095, section 5.3.1.). Holding time, 180 days.

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4.3.2. **TKN working calibration standard (CAL STD, 4 mg N/L)** - To a 1 L volumetric flask containing approximately 600 mL DI water add 2.0 mL conc. H_2SO_4 (4.1.5., final acid concentration 0.2 % (v/v)) followed by 4.0 mL NH_3 stock standard (1000 mg N/L, 4.3.1.) and fill up to the mark with DI water. Invert 12 times to mix. Store this standard at room temperature and digest according to DEP SOP NU-091. Holding time, 30 days.

4.3.3. **CCV (1 mg N/L)** - To a 1 L volumetric flask containing approximately 600 mL DI water add 2.0 mL conc. H_2SO_4 (4.1.5.) followed by 1.0 mL NH_3 stock standard (1000 mg N/L, 4.3.1.) and fill up to the mark with DI water. Invert 12 times to mix. Store this standard at room temperature and digest according to DEP SOP NU-091. Holding time, 30 days.

4.3.4. **ALTQC standard** - The quality control standard (4.1.14.) is prepared from the stock standard according to the manufacturer's instructions and digested according to DEP SOP NU-091. The supplier for the QC stock standards (e.g. Environmental Resource Associates) differs from the supplier of the calibration standards. The stock ALTQC standard may be diluted appropriately so that the concentrations falls within the calibration range. For the concentration and expiration date of the ampule see the manufacturer's certificate. Once prepared according to the manufacturer's instructions, the stock solution has a holding time of 90 days. If diluted further as a working solution, the holding time is 30 days.

4.3.5. **TKN/TP Organic Spiking Stock Solution (TKN: 150 mg N/L, TP: 50 mg P/L)** - Dry approx. 1.1 g glycine (4.1.3.) overnight at 60 °C. Add 0.8040 g glycine and 0.4941 g β-glycerophosphoric acid (4.1.12.) to a 1 L volumetric flask containing approx. 400 mL DI water. Bring to volume with DI water. Invert 12 times. Do not acidify. Store in refrigerator. Holding time, 180 days.
This solution is used to prepare laboratory fortified blanks (LFB), matrix spikes (MS) and matrix spike duplicates (MSD). This solution is also used for the analysis of TP and TKN in soils (DEP SOPS NU-084 and NU-081, respectively).

Note: **Glycine:** $C_2H_5NO_2$, Formula weight: 75.07 g/mol.
Molecular weight factor = 14.0067/75.07 = 0.1866.
β-glycerophosphoric acid: $C_3O_{11}H_{17}PNa_2$, Formula weight: 306.11 g/mol. Molecular weight factor = 30.9738/306.11 = 0.1012.

4.3.6. **TKN/TP PQL standard (PQCK2) (TKN: 0.20 mg N/L, TP: 0.06 mg P/L)** - To a 1 L volumetric flask, containing about 500 mL of DI water, add 200 µL of NH_3 stock standard (1000 mg N/L, 4.3.1), 60 µL of TP stock standard (1000 mg P/L, see DEP SOP NU-084, section 4.3.1.) and 2 mL of conc. H_2SO_4 (final acid concentration 0.2 % (v/v)). Bring to volume with DI water. Digest this standard according to DEP SOP NU-

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091. This solution is also used for the analysis of TP and TKN in soils (DEP SOP NU-084 and NU-081, respectively). Holding time, 30 days.

5. SAMPLE HANDLING AND PREPARATION

Refer to DEP SOP NU-091 for preparation, sample preservation, and handling details.

6. SAMPLE ANALYSIS AND QUANTITATION (SEE DEP SOP NU-033)

Note: • Digested solutions containing particulate matter should be filtered through a 0.45 μm filter prior to analysis on the AQ2 Discrete Analyzer and then redigested with dilution to confirm the result.
• Samples with organic N concentrations above 10 mg N/L must be diluted prior to digestion in order to report results without a J qualifier. (Subtract the ammonia concentration, if known, from the TKN result to calculate the organic N concentration). Add a comment on the run report to document this observation.
• Observe routine maintenance as recommended by the instrument manufacturer and enter any maintenance performed in the maintenance logbook.

6.1. Initial Preparation:

6.1.1. Remove reagents from the refrigerator. Check the expiration dates on all standards and reagents. Discard expired standards and/or reagents and reprepare if necessary.

6.1.2. Select digested trays to be analyzed and vortex samples and standards thoroughly. Record worksheet ID/tray number on the Reagent and Standard Logsheet.

6.2. Calibration:

6.2.1. The instrument is calibrated using the digested 4.0 mg N/L working CAL STD (4.3.2).

6.2.2. The diluent for TKN is the digested blank (DEP SOP NU-091, 5.3.1.).

6.2.3. The auto calibration feature of the AQ2 is used in the analysis of TKN. The standards in the calibration are as follows (C2 to C6 are prepared by the instrument from the digested working calibration solution (4.3.2.)).

STD	Conc. (mg N/L)	Source
C1	0.00	Digested blank (6.2.2.)
C2	0.20	Instrument dilution of C7
C3	0.40	Instrument dilution of C7
C4	1.0	Instrument dilution of C7

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C5	2.0	Instrument dilution of C7
C6	3.0	Instrument dilution of C7
C7	4.0	Digested CAL STD (4.3.2)

6.3. Reagents, Controls, and Stock Standard Loading:

6.3.1. Fill three separately labeled AQ2 reagent vessels with the following: buffer (4.2.5.), salicylate (4.2.4.3.) and hypochlorite (4.2.6.).

6.3.2. Reagent Location - The reagent tray, with plastic wedge shaped containers is located on the right side of the injector arm. It holds all reagents and controls (CCV and blank) in the assigned locations. The buffer is in position 4, the salicylate reagent in position 5, and the hypochlorite reagent is in position 6.

6.3.3. Controls - Fill one reagent vessel with the digested 1.0 mg N/L CCV (4.3.3.) and place it in position 10. Fill another vessel with digested blank (CCB, 6.2.2.) and place it in position 11.

6.4. Analysis Setup/Sequence setup:

6.4.1. Turn on the AQ2 Analyzer (large button on the right side at the back of the instrument) and the computer. To start the software double click on the AQ2 Icon on the screen. The username is '**Instrument 1'** or **'Instrument 2'** and the password is 'password'.

6.4.2. Check the DI water reservoir and the waste containers. Replace all used reaction segments.

6.4.3. In the software, go to the 'Settings' menu and select maintenance. Zero the reaction segment counter.

6.4.4. In the 'settings' menu, select the daily start-up option.

6.4.5. Setup of the analytical run - Click on the scheduling icon on the left side of the screen. Choose **Reagent Set 3**. Select a free tray and name the run using the format TKNYYYYMMDD001K or Q (K for instrument 1, Q for instrument 2). For the first run on instrument 1 on 03/24/2014 the name of the run would be TKN20140324001K. The sequence screen should appear. Starting at line 3, type the sample sequence in the 'Sample ID' column. Include at least 7 blank "C" cups at the end of the run to avoid a known timing issue affecting the final CCB/CCV results (see the Seal AQ2 Method EPA-111-A, Rev. 4 for more information). Highlight the typed lines and select the **TKN** test. The test should appear in the requested test column to the right of the sample ID's. Click the 'save'

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icon. Click on the 'print' icon to print the bench sheet.

A typical run sequence includes the following:

CCV (4.3.3.) and **CCB** (digested blank, 6.2.2.) – these checks are scheduled to bracket every ten cups on the run)
PQCK2 (4.3.6.)
Batch **MBLKs** and **LFBs**
ERAQC# (ALTQC, 4.3.4.)
Spiked samples and **spike duplicates** (4.3.5.)
Field and **Equipment blanks** from batched samples
Batched samples
Final **CCV/CCB** set
Seven blank "C" cups

Note: The AQ2 run sequence is set up to run batch QC before batched samples in order to screen for problems, as digested chemistries require quick decisions in the event of failing QC.

6.4.6. Pouring the analytical run - Cup positions one and two are reserved for the calibration standards. The TKN calibration solution (6.2.1.) is placed in cup two. Pour the samples according to the cup position on the bench sheet.

6.4.7. Double-click on the 'Run' icon on the left side of the screen. A dialog box will appear. Select the tray that contains the sequence to be analyzed. Click 'OK'. Another dialog box will appear. Check the auto-standardize box and click 'OK'. (If the analysis should use the previous calibration do not check the auto-standardize option.) Select 'OK' on the following dialog box.

6.4.8. The reagent levels are checked by the instrument and are displayed on the upper right corner of your screen. If the levels are sufficient for the entire run the analysis will start; if not, the run will stop and display the reagent which has insufficient volume. Fill up the reagent and re-start the run according to 6.4.7.

6.5. Quantitation/Calculations:

6.5.1. The instrument will give the analysis results for TKN in mg N/L.

6.5.2. The QC-Manager calculates recoveries for spikes and QC standards as well as relative percent differences (RPD) for duplicate samples and spikes. For general calculations including RPDs and spike recoveries see Section 5.9 of the FDEP Quality Manual.

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6.5.3. The high calibration standard is prepared according to section 4.3.2. The other standards used in the second order calibration are auto-diluted by the instrument from the high standard. The correlation coefficient for the calibration is calculated by the instrument software.

6.6. Post-Run Procedure:

6.6.1. After the run has finished, print the AQ2 Test Parameters Report from the 'Settings' menu on the top of the screen.

6.6.2. Click on the 'Acceptance' menu located on the left side of the screen.

6.6.3. Click on the 'Accept All' Icon. This adds 'ACC' to the results. A pop-up window appears where the file name needs to be entered. Follow the naming convention described in 6.4.5. Accepting the run in the AQ2 creates the excel transfer file for uploading the data to the Laboratory Information Management System (LIMS). This file should be saved in the current year's subfolder located in \\FLDEP1\DEAR\Labs\lab3\Chem_Data\Inorganic\Nutrients\AQ2\TKN_COP\.

6.6.4. Convert the excel file into Raw data in the QC Manager. The Instrument used is 'Nutrients: Seal AQ2 1' or 'Nutrients: Seal AQ2 2'.

6.6.5. Click on the 'Reporting' icon on the left side of the screen. Check the reporting format 'TKN/TP' and print the run report. It contains the calibration and all the data. Select the chronological order report function to print your report.

7. DATA ARCHIVAL

7.1. The raw data is archived in paper format. A report folder is created for each analytical run. Occasionally more than one run may be in one folder. The folder is labeled with the test (W-TKN, W-TKN-F), filename/s (TKNYYYYMMDD001K or Q), and initials of the analyst.

7.2. Each report folder should contain the following items - run report including raw data, calibration curve, method parameters, bench sheet, Reagent and Standard Logsheet, digestion temperature data logger report, and a copy of the data and QC assessments uploaded from QC Manager to LIMS at the time of test authorization. The sample preparation worksheet is archived electronically in the Prep Worksheet Manager module in the LIMS system.

7.3. The report folders are on file with the nutrients and general chemistry supervisory personnel. Periodically, the files are moved to the State Records Center for long time storage according to DEP SOP LB-006.

7.4. AQ2 instrument data excel transfer files for upload to QC manager are stored in

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electronic format in the current year's subfolder located in: \\FLDEP1\DEAR\Labs\lab3\Chem_Data\Inorganic\Nutrients\AQ2\TKN_COP\.

8. QUALITY CONTROL/DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES (SEE NU-038 AND NU-043)

8.1. For method performance and acceptance criteria for QC measures and corrective action for out-of-control data and contingencies for handling out-of-control or unacceptable data refer to DEP SOPS NU-038 and NU-043.

8.2. A CCV (4.3.3.) and a CCB (digested blank, DEP SOP NU-091, 5.3.1.) are analyzed every 10 samples and at the beginning and end of each run. Recovery acceptance limits for the method blanks (MBLKs) and CCBs are < 0.080 and > -0.080 mg N/L. The check standard (CCV) recovery limits are 90 – 110 %

8.3. Each tray of digested samples contains a digested PQL level standard (PQCK2, 4.3.6.) and a certified standard reference material (ALTQC, 4.3.4.). Recovery acceptance limits are 70 -130 % for PQCK2 and 90 – 110 % for the ALTQC.

8.4. An MBLK, an LFB, a duplicate sample and/or duplicate matrix spikes are digested and analyzed with every batch of 10 or fewer samples (see DEP SOP NU-091). Acceptance limits for the LFB and matrix spike recoveries are 90 – 110 %. The RPD of sample/matrix spike duplicates must be less than 20 % for results over the PQL.

8.5. Linear Calibration Range (LCR) - See FDEP Quality Manual, Appendix B, Section 7.0, 'Protocol for Determining the Test Method Range of Applicability'. To verify the calibration range the recoveries for calibration standards above the PQL must be 90 -110 %. The correlation coefficient of the second order calibration must be larger than 0.995. Every day samples are analyzed a new calibration has to be performed and verified at the beginning of the analysis. Consecutive runs on one day may use the same calibration.

9. SAFETY/HAZARDOUS WASTE MANAGEMENT

9.1. Review the Laboratory Health and Safety Plan (accessible on the intranet at https://www.fldepnet.org/system/files/Division-of-Environmental-Assessment-and-Restoration/governing-documents/Health%20%20Safety%20Plan_0.docx).

9.2. Safety glasses, gloves and lab coats should be worn at all times whilst handling samples and reagents.

9.3. Read the MSDS information for safe reagent and sample handling.

9.4. The solution used to digest samples contains copper, which is a hazardous waste and toxic. All waste from the instrument, digested samples, and standards made

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with digestion solution should be collected in bottles clearly labeled as hazardous waste. The waste should be disposed as acidic metals waste.

10. REFERENCES

For definitions see section 3 of the FDEP Quality Manual. All abbreviations used throughout the document are defined at least once in the document usually at the first time of use. Method validation data is on file with the supervisor.

10.1. EPA Method 351.2, Rev. 2.0 (1993).

10.2. Seal AQ2 Method EPA-111-A, Rev. 4, Total Kjeldahl Nitrogen-N (Copper Catalyst) in Drinking, Ground and Surface Waters, Domestic and Industrial Wastes.

10.3. DEP SOP NU-033, Summary of Analysis Procedure for the General Chemistry and Nutrients Workgroups.

10.4. DEP SOP NU-038, Nutrients and General Chemistry: Quality Assurance and Quality Control.

10.5. DEP SOP NU-041, Reagent Preparation Procedure.

10.6. DEP SOP NU-042, Standard Preparation Procedure.

10.7. DEP SOP NU-043, Nutrients and General Chemistry Quality Assurance and Data Authorization Guidelines.

10.8. DEP SOP NU-091, Preparation of Waters for Total Kjeldahl Nitrogen Analysis (Copper Catalyst).

10.9. DEP SOP NU-081, Determination of Total Kjeldahl Nitrogen in Soils using the Westco SmartChem Discrete Analyzer.

10.10. DEP SOP NU-084, Determination of Total Phosphorus in Soils using the AQ2 Discrete Analyzer.

10.11. DEP SOP NU-095, Determination of Ammonia in Water by Gas Diffusion Segmented Flow Analysis.

10.12. DEP SOP LB-006, Standard Operating Procedure for Records Maintenance and Storage.

10.13. Quality Manual, Bureau of Laboratories, FDEP.

10.14. Laboratory Health and Safety Plan.

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Appendix of Significant Changes

10/20/2009 Some minor corrections were made in section 4.

08/20/2010 Procedural clarifications some corrections mainly to section 4 and 6.

08/09/2011 Preparations of the PQL standard and spiking TKN solution were modified and now include phosphorous (these solutions also used for TKN and TP analysis in soils).

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