PREPARATION OF WATERS FOR TOTAL KJELDAHL NITROGEN ANALYSIS (COPPER CATALYST)

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1. SCOPE AND APPLICATION

1.1. This method details the digestion of waters for Total Kjeldahl Nitrogen (TKN) analysis. Digestates produced by this method are suitable for analysis by the Seal AQ2 Discrete Analyzer (DEP SOP NU-092). For analytical details such as calibration range and the method detection limit (MDL) see DEP SOP NU-092.

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

1.3. Applicable matrices are surface and saline water, and domestic and industrial wastewater.

2. SUMMARY OF THE METHOD

2.1. This SOP is based on EPA method number 351.2, Rev. 2.0 (1993) and AQ2 method No: EPA-111-A Rev.4

2.2. A digestion solution containing potassium sulfate, sulfuric acid and copper sulfate catalyst is added to the samples. They are heated in a digestion block to convert organic nitrogen to its inorganic form (NH_4^+). The potassium sulfate raises the boiling temperature of the digestion and increases the rate of conversion.

2.3. Interferences: Nitrate in concentrations greater than 10 mg N/L can interfere with the digestion of TKN. 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 must be reported 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. Equipment:

Equipment	Manufacturer/Vendor[1]	Model/Catalog Number
Digestion Block	SEAL Analytical	BD50
Type K Thermocouple	Omega Engineering	TJ72-CAXL-316U
Temperature Data Logger	Fisher Scientific	15-059-299
Male Connector	Omega Engineering	NC1658189
Vortex Mixer	Corning	LSE
Analytical Balance	Mettler Toledo	MS204TS/00
Stirrer/Hot Plate	Corning	PC-420D
Stir bar	Fisher Scientific (SR)	14-513-61
Drying Oven	ThermoScientific	Lindberg Blue M
Desiccator Cabinet	Fisherbrand	08-645-6
Adjustable Pipette, 10–100 µL	Eppendorf/Fisher	Research Plus, 3123000047
Adjustable Pipette, 100–1000 µL	Eppendorf/Fisher	Research Plus, 3123000063
Adjustable Pipette, 2-20 mL	Rainin	LTS L-20MLXLS, 17011788
Bottletop Dispenser, 0.5-5 mL	BrandTech	Dispensette S, 4600130
Bottletop Dispenser, 2.5-25 mL	Eppendorf/Fisher	Varispenser 2, 4966000045
Digestion Tubes, 75 mL	SEAL Analytical	4003005
Digestion Beads, 0.25”	Saint-Gobain	09-191-30B

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Poxygrid Test Tube Racks	Bel-Art	F18764-0001
Volumetric Flask, Class A, Assorted	Fisher (SR)	10-212B; 10-212E; 10-212-5L; 10-208M
Glass Beaker, Assorted	Fisher (SR)	02-540G; 02-540K; 02-540M; 02-555-20E; 20-540R
Graduated Cylinder, Class A	Fisher (SR)	08-564-A
Or Equivalent [1]**Note:** (SR) designates equipment is typically ordered through the in-house stockroom		

3.2 Consumables and frequently replaced supplies:

Supplies/Consumables	Vendor[1]	Catalog Number
Pipette tips, Eppendorf, 200 µL	Fisher (SR)	05-403-41
Pipette tips, Eppendorf, 1000 µL	Fisher (SR)	05-403-43
Pipette tips, Rainin, 20 mL	Rainin (SR)	17001131
Sample Beakers, Polypropylene, Assorted	Fisher (SR)	08-732-119; 08-732-120; 08-732-122
Kimwipes, Assorted	Fisher (SR)	06-666A; 06-666C
Weigh Dish, Hexagonal	Fisher (SR)	02-202-102
Weigh Dish, Aluminum, 110 mm	Fisher (SR)	08-732-100
Parafilm	Fisher (SR)	13-374-12
Aluminum Foil	Fisher (SR)	01-213-105
Or Equivalent [1]**Note:** (SR) designates supply is typically ordered through the in-house stockroom		

Note: Each data logger and thermocouple combination is calibrated annually as a system, and the thermocouples and data loggers cannot be interchanged. Calibration certificates and system information are on file with the Nutrients supervisors. One USB interface cable allows all MadgeTech data logger devices to be connected to a PC running the MadgeTech4 software to start and stop recording, download temperature data, etc.

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

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.

Note: 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. If available, a Certificate of Analysis for each purchased reagent or standard is also maintained in the REAGENT_PREP folder on the Nutrients drive. All containers holding standards or reagents must be labeled with their unique ID and expiration date.

Note: Prior to using the chemicals in this analysis, read their MSDS sheet and the safety notes found in Section 8 of this SOP.

4.1. Chemicals:

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	Chemical	Alias	Manufacturer[1]	Catalog Number	Purity	Expiration[2]	Storage
4.1.1.	DI water	H_2O	In house	N/A	N/A	Daily	N/A
4.1.2.	Potassium sulfate	K_2SO_4	Fisher Chemical (SR)	P305-500	ACS, ≥99%	See COA	Room Temp
4.1.3.	Cupric sulfate pentahydrate	$CuSO_4$ *$5H_2O$	Fisher Chemical	C493-500	ACS, ≥98%	See COA	Room Temp
4.1.4.	Sulfuric acid	H_2SO_4	Fisher Chemical (SR)	A300S-212	Concentrated, ACS Plus ≥95%	See COA	Room Temp
4.1.5.	Hydrochloric Acid	HCl, For cleaning	Fisher Chemical (SR)	A144S-212	Concentrated, ACS Plus	See COA	Room Temp
4.1.6.	Nitric Acid	HNO_3, For cleaning	Fisher Chemical (SR)	A509P-212	Concentrated, Trace Metal	See COA	Room Temp
4.1.7.	Ottawa Sand	SiO_2, For probes	Fisher Chemical (SR)	S23-3	N/A	See COA	Room Temp
Or Equivalent [1]**Note:** (SR) designates chemical is typically ordered through the in-house stockroom [2]**Note:** (*) designates manufacturer's suggested retest date. No expiration date given.							

Note: Wash all glassware and bottles with 1:1 HCl and rinse at least 7 times with DI water before preparing reagents and standards. Make sure any containers that are used to store prepared reagents and standards are clean and dry.

4.2. Reagents:

4.2.1. Diluent –

Sulfuric acid (4.1.4.)	4 mL
DI water (4.1.1.)	to 2000 mL

In a 2 L volumetric flask containing approx. 1.8 L of DI water, pipette 4 mL of sulfuric acid. Bring to volume with DI water and invert to mix thoroughly.

Store at room temperature. Holding time, 30 days.

4.2.2. Copper Sulfate Digestion Solution –

Sulfuric acid (4.1.4.)	268 mL
Potassium sulfate (4.1.2.)	268 g
Cupric sulfate pentahydrate (4.1.3.)	22.8 g
DI water (4.1.1.)	to 2000 mL

Place a 2 L beaker filled with approx. 1.2 L of DI water and a stir bar on a stir plate in a fume hood. Slowly add 268 mL of sulfuric acid while stirring. Then dissolve 268 g of potassium sulfate and 22.8 g of copper sulfate. Let solution stir until it reaches room temperature, then transfer to a 2 L volumetric flask. Bring to volume with DI water and invert to mix thoroughly.

Caution: This solution will become hot. If needed, cover with parafilm after letting cool slightly and let come to room temperature overnight. Make sure that the beaker is clearly labeled.

Transfer to clean glass bottle. Store at room temperature. Holding time, 30 days.

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4.3. Standards and spiking solution:

For detailed preparation instructions please refer to the DEP SOP NU-049 and NU-092.

4.3.1. TKN/TP PQL standard (PQCK2) –

TKN: 0.20 mg N/L, TP: 0.06 mg P/L

See DEP SOP NU-092, 4.3.6. for preparation instructions.

4.3.2. TKN/TP Organic Spiking Solution –

TKN: 150 mg N/L, TP: 50 mg P/L

See DEP SOP NU-092, 4.3.5. for preparation instructions.

4.3.3. ALTQC standard –

The ALTQC standard is used as a second source standard and must have a different supplier or a different lot number than the calibration standards. A certified reference standard is preferred. Currently, the second source supplier used is Environmental Resource Associates (ERA).

TKN: Refer to Certificate of Analysis (COA)

See DEP SOP NU-092, 4.3.4. for preparation instructions.

4.3.4. TKN Working Calibration Standard (CAL STD) –

TKN: 4.0 mg N/L

See DEP SOP NU-092, 4.3.2. for preparation instructions.

4.3.5. TKN Continuing Calibration Verification (CCV) Standard –

TKN: 1.0 mg N/L

See DEP SOP NU-092, 4.3.3. for preparation instructions.

5. SAMPLE PRESERVATION, PREPARATION AND HANDLING (SEE DEP SOP NU-033)

5.1. Aqueous samples are preserved at collection with sulfuric acid to a pH < 2 (0.2 % sulfuric acid). They are stored upon receipt in the Nutrient refrigerator located in the receiving room at ≤ 6 °C (not frozen) and have a 28-day holding time. Remove the samples from the refrigerator following the proper protocols to document the sample chain of custody. Allow samples to come to room temperature before digestion. Begin heating the digestion block to 180 °C using the appropriate program on the block controller (see section 5.4.10 below).

5.2. While preparing the samples any extra sample waste not containing copper can be discarded down the sink. Any samples/solutions containing digestion solution that need to be discarded are to be collected in a hazardous waste container.

5.3. The following is required to setup a tray for digestion:

Note: All Samples having conductivities above 5000 umhos/cm (equivalent to 3.0 ppt salinity) are batched separately and diluted two-fold prior to digestion. If a sample conductivity exceeds

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5000 umhos/cm it should be digested in a salt batch even if it is not labeled as a salt matrix. Samples with organic N concentrations above 10 mg N/L must be diluted prior to digestion 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.

5.3.1. One blank (MBLK) for each batch (up to10 samples) - the diluent (4.2.1.) is poured into the digestion tube.

5.3.2. One PQCK2 (4.3.1.) for each tray.

5.3.3. One ALTQC (4.3.3.) for each tray.

5.3.4. One laboratory fortified blank (LFB) per batch. The diluent is spiked at a concentration of 1.2 mg N/L TKN by adding 200 µL of 150 mg N/L TKN spiking solution (4.3.2.) to 25 mL diluent (4.2.1.) in the digestion tube.

5.3.5. One matrix spike per batch (1.2 mg N/L). Prepare by adding 200 µL of 150 mg N/L TKN spiking solution (4.3.2.) to 25 mL of sample (diluted if needed) in the digestion tube.

5.3.6. One duplicate sample and/or duplicate matrix spikes for each batch.

5.3.7. Samples, including samples to be re-digested.

5.3.8. Additional QC as needed, typically CCV standard (4.3.5.), blanks, and working calibration standard (4.3.4.).

5.3.9. The appropriate prep worksheet (see section 5.4.3.) which contains the prep batch ID numbers and other batching information for the samples, and the temperature report for the digestion (see section 5.4.10.1. f.).

5.4. Preparation Procedure for Water Samples:

Note: For the preparation of all dilutions use the diluent, 0.2 % H_2SO_4 (4.2.1.).

5.4.1. Record refrigerator temperature before opening door. Remove the following standard solutions from the refrigerator: spiking solution (4.3.2.), PQL solution (4.3.1.), ALTQC solution (4.3.3.), CAL STD (4.3.4.), and the CCV check solution (4.3.5.). Allow standards to come to room temperature before pouring them out for digestion.

5.4.2. For each tray one PQCK2, ALTQC, CAL STD and CCV check standard should accompany the batches. For every batch digested, a blank (MBLK), an LFB, a matrix spike and a duplicate sample and/or duplicate matrix spike are required.

5.4.3. Create a new prep worksheet in the Prep Worksheet Manager module in LIMS:

5.4.3.1. Open the Prep Worksheet Manager module in LIMS and choose the first icon (which looks like a blank page) to create a new worksheet. Choose “351.2 (March 1993)” from the list of available preparation IDs, select the sample IDs to be added to the worksheet, assign the worksheet to the correct analyst, and click the checkmark to create the

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worksheet. The worksheet ID generated in LIMS will be the tray number for the digestion.

5.4.3.2. Open the new worksheet by clicking the spreadsheet icon in the Prep Worksheet Manager module and double-clicking the appropriate worksheet ID from the list. Complete the header information for the sheet: SOP (NU-091), prep analyst name, prep start date/time and prep completion date/time, equipment ID (e.g., digestion block 'Thunderbird', 'Hercules', or 'Goliath'), pipettes and dispensers used, and the reagents and spike standard solution serial numbers (ensure that the corresponding identities and expiration dates are correct by comparing to the labels on the bottles and equipment). The prep start time should be reflected by the initial time on the datalogger upon activation, while the completion time should be reflected by the time of the final data point that the datalogger obtains.

5.4.3.3. Update the sample table, beginning with the starter rows for QC samples digested on the tray, ensure that serial numbers, identities, and expiration dates for the standard solutions used are correct. A single prep batch ID will be generated automatically for every sample on the sheet; therefore, assign other prep batch IDs, as necessary, by 'Control+Clicking' the sample IDs, MBLK, and LFB to be batched together and generating a new prep batch ID for each batch. For the matrix spike sample in each batch, duplicate its row in the sample table **twice** by right-clicking on the row and selecting "Duplicate Row", then add the spike code '_O1_' and the letter A or B as a suffix to the sample ID. Save the updates by clicking the "Save" button at the bottom of the worksheet.

5.4.3.4. Once the information in the worksheet is complete and correct (see section 5.4.6. regarding dilution factors and sample volumes), ask a supervisor to review the worksheet.

5.4.4. Dry the outside of the clean tubes. If no clean tubes are available, clean the number of tubes needed before proceeding. The detailed procedure for cleaning tubes is given in DEP SOP NU-046.

5.4.5. Label the tubes using a black permanent marker following the batching order on the digestion worksheet. The worksheet ID/tray number is written on the first tube on the tray.

Note: Tube markings made with a black marker are known to survive the harsh conditions of the digestion. Markers of other colors may not produce stable enough markings.

5.4.6. If the tubes were cleaned manually rinse them with DI water 10 times. Pour out any excess water in the clean tubes before pouring in the sample. Shake each sample well, and then rinse each tube with 1-2 mL of sample. For dishwasher-cleaned tubes this practice usually is not required.

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Pipet 25 mL of each sample into the respective tubes. If sample requires dilution, the dilution can be prepared either in a digestion tube or in a volumetric flask.

For example, if a sample requires 1:2 dilution prior to digestion, pipet 12.5 mL of the sample into the digestion tube and add 12.5 mL of Diluent (4.2.1). For higher dilution factors, use a volumetric flask to prepare the dilution by pipetting the appropriate amount of sample into the flask first and then bring the volume up to the mark with Diluent (4.2.1); transfer 25 mL of the diluted sample into the digestion tube.

Note: NEVER dilute Equipment Blanks, Field Blanks or Trip Blanks samples unless told to do so by a supervisor.

Record dilution factors and actual sample volume used on the prep worksheet.

Pour extra blanks using the diluent for dilutions and continuing calibration blanks (CCB) on the analytical run.

5.4.7. Add 2 clean beads to each tube. Do not add more than 3 beads to water samples.

5.4.8. Add 5 mL of the copper sulfate digestion solution (4.2.2.) to each tube.

5.4.9. Place the tubes in the digestion block.

5.4.10. One spot on the block is chosen to place the thermocouple of the data logger system (3.10.). This system is used for temperature recording during the digestion process. The data logger program is started in advance using the MadgeTech4 software, as follows:

a. Plug data logger into the USB cable at the PC with the MadgeTech4 software installed.

b. Open the MadgeTech4 software. The data logger ID will appear in the "Connected Devices" panel of the software interface. Click on the ID once to highlight it.

c. In the Device tab menu bar, click Quick Start (this will use the most recent device settings, visible in the Connected Devices panel). When the data logger has been properly started, the status will read "Running". It is recommended that data collection rate is set to 1 point every 5 minutes.

d. Unplug the data logger from the USB interface cable, and attach to the appropriate thermocouple cable outside the hood where the digestion block sits.

e. After the digestion is complete (see section 5.4.13.), unplug the thermocouple cable and repeat steps a and b, above. Click on the probe entry that appears and click "Stop" in the Device tab.

f. Click "Download" to open the temperature report. Go to the Report tab and click on "Generate" button to generate data table.

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This will create a data table tab. Right click on the data table tab and select "Rename." Use the worksheet ID/tray number and Probe ID to name the temperature file in the dialog box that opens. Use Save To option to save the report on the Chemistry drive at \\fldep1\dear\Labs\Lab5\Chemistry\InorganicPrep\WRKSHTS\Templates\CURRENT\Nutrients\TKN-HighTP\Temperature logs. Print the report, which is to be included in the run folder.

g. Close the MadgeTech4 software and recharge the data logger battery if necessary.

5.4.11. Select and execute the block digestion program.

5.4.11.1. If the block was preheated to 180 °C using program 2, continue with program 3. Program 3 holds the temperature at 180 °C for 1 hour, after which it ramps up to 380 °C at 5 °C/min, and hold at that temperature for 1 hour. The block controller settings may be adjusted depending upon the actual temperature recorded by the data logger-thermocouple system; block temperatures should be verified whenever the controller settings are adjusted and documented in the block maintenance manual located in B403.

Note: The method requirement for complete digestion is a minimum of 30 min at 380 °C. Block temperatures above 395 °C should be avoided due to the possibility of side reactions.

5.4.11.2. If the block was not preheated at the time the tubes were placed in the block start the digestion by selecting program 3 (it will start by ramping up to 180 °C and then continue as described above). These initial heating stages are necessary to hydrolyze the organic nitrogen and drive off most of the water. Watch the samples to be sure they do not "bump" out of the tubes.

5.4.12. Return the ALTQC, spiking, CCV, PQL, and calibration standard solutions to the refrigerator.

5.4.13. Check samples back into the receiving room.

5.4.14. Once the digestion is complete carefully disconnect the data logger and place the tubes in a metal rack and allow the samples to cool for approximately 15-20 minutes under the hood.

5.4.15. Follow the instructions in 5.4.10. to download temperature data from the data logger. Ensure that temperature of the digestion stayed continuously above 380 C^{o} for at least 30 min.

5.4.16. Once the samples are cool enough to handle, use a pipette or bottle-top dispenser to add 25 mL of DI-Water.

Note: Before removing tubes from block be prepared with heat resistant gloves in the event of a tube falling through the holding tray.

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WARNING: The samples at this point consist of a concentrated sulfuric acid/salt mixture. The addition of DI water may generate much heat and thus poses a significant hazard. Be sure to point the tube away from your face and swirl gently to mix.

5.4.17. Parafilm the sample tubes individually and vortex until tube contents are fully mixed.

5.4.18. Check the bottom inside of the tubes for any precipitation. Salt samples are sometimes difficult to mix homogeneously because of the precipitation. If there is any difficulty getting the sample to mix well, remove the parafilm and slightly reheat the tube(s) in a water bath. Vortex and parafilm again. Digested solutions containing particulate matter should be filtered through a 0.45 μm filter prior to analysis on the AQ2 Discrete Analyzer and then re-digested with dilution report the result.

5.4.19. The samples are now ready for analysis.

After the prep worksheet has been reviewed and any necessary notes, changes, or additions have been saved, complete the worksheet by clicking the "Complete" button in the lower left corner of the worksheet – this changes the status of the samples from "W" to "V" in LIMS. Active or completed worksheets can be printed or exported in excel format for convenience when setting up the AQ2 analysis.

Make sure the worksheet ID/tray number is marked clearly on the first tube in the tray, as well as on the data logger temperature report. The data logger temperature report is printed and submitted to the analyst for inclusion with the analysis folder. The digestion worksheet itself is reviewed in LIMS directly and archived as part of the LIMS program.

6. SAMPLE ANALYSIS/QUANTITATION/DATA ARCHIVAL

Refer to the DEP SOP NU-092 for analysis procedure, quantitation information, data archival, and method validation.

7. QUALITY CONTROL (SEE DEP SOPS NU-038 AND NU-043)

Samples with similar background matrices are grouped as a digestion batch and prepared together. A digestion batch is defined as a set of 10 samples or less from the same matrix group. The following are possible matrices that can be grouped together as a matrix group; each is prepared with one set of quality control samples. If a sample or group of samples is visibly different from the rest, they should be batched separately.

Matrix Group	Matrices grouped together as one batch
A	W-GROUND, W-SURF-FRH, and W-DRINKING
B	Conductivity > 5000 umhos/cm; 1:2 predilution
C	W-EFFLUENT, W-INFLUENT

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Samples with the matrix WATER should be batched with Group A unless the conductivity is greater than 5000 umhos/cm in which case they should be batched with Group B.

7.1. Digested PQCK2 - one per tray.

7.2. Digested standard reference material (ALTQC) - one per tray.

7.3. Digested LFB - one per batch.

7.4. Digested method Blank - one per batch.

7.5. Select samples for quality control - Randomly choose a sample to be used as the quality control sample for each batch. This sample may be changed if upon observation it seems not to be a representative of the rest of the samples e.g., presence of a great deal of particulate or much different color. In this case you may treat this sample as a separate batch and continue with the sample preparation.

7.6. Sample matrix spike/s - Using the sample selected as QC sample (7.5.), prepare and digest a matrix spike or a set of duplicate spikes for each batch. If only one matrix spike is prepared the QC sample must be digested in duplicate.

7.7. 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 the following DEP SOP'S: NU-092 and NU-043.

8. EQUIPMENT MAINTENANCE

8.1. Maintenance of the fume hoods and digestion blocks should be performed, at least, after every five uses. Maintenance must be documented in the block maintenance logbook. The maintenance for the fume hood is recorded in the logbook of the digestion block that is housed inside it.

8.2. The maintenance of both the fume hoods and digestion blocks include, but are not limited to:

8.2.1. The cleaning and wiping down of any dust and condensed sulfuric acid that has accumulated in the hoods.

8.2.2. The cleaning and wiping down of the digestion block to remove any corrosion or condensed sulfuric acid accumulation.

8.2.3. The replacement of bench paper that lines the bottom of the hood

8.2.4. The inspection and cleaning of the temperature probe that is being used with each block.

9. SAFETY/HAZARDOUS WASTE MANAGEMENT/POLLUTION PREVENTION

9.1. Review the Laboratory Health and Safety Plan (accessible on the intranet at https://www.fldepnet.org/content/dear/bureau-laboratories-safety-documents).

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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 sample waste that contains digestion solution (i.e., prior to or after digestion) should also be collected in bottles labeled as 'Hazardous Waste,' and disposed as acidic metals waste. No more than three full bottles of digestion solution waste should be stored in the prep lab. A full waste bottle should not be stored more than three days in the lab.

9.5 Any concentrated acid used in the lab must be diluted down before it can be disposed of down the sink. For it to be considered tolerable for sink waste, it must be diluted to 2% or by a dilution factor of 50.

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.

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 Sample Analysis Procedures 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-046, Procedure for TKN/TP/LLTP Glassware Cleaning.

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-092, Determination of Total Kjeldahl Nitrogen in Water using the Seal AQ2 Discrete Analyzer

10.12. Quality Manual, Bureau of Laboratories, FDEP.

10.13. Laboratory Health and Safety Plan.

10.14. DEP SOP LB-006, Standard operating procedure for records maintenance and storage.

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Appendix of Significant Changes

08/20/2010	Clarify instructions on procedure and requirements for the digestion of high conductivity samples (Sections 4, 5, and 7).
08/12/2014	Instructions are added for the use of the temperature logger systems and the preparation worksheet manager module within the LIMS program. (Section 3.10 and 5.4)
05/06/2015	Use of pipettes to transfer sample aliquots into the digestion tubes. (Section 5.4.6. and 5.4.13)
07/26/2018	Section 8. **EQUIPMENT MAINTENANCE** – was added**.**
12/22/2020	In Section 3, the equipment and supplies were updated and inputted into table format. In Section 4, chemicals were updated and inputted into table format. Preparation procedures for reagents and standards were reformatted and clarified.

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