ANALYSIS OF LOW-LEVEL TOTAL NITROGEN (TN) AND DISSOLVED NITROGEN (DN) USING THE SHIMADZU TOC-L2 INSTRUMENT EQUIPPED WITH A TOTAL NITROGEN MODULE (TNM-L)

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1. SCOPE AND APPLICATION

1.1. This SOP details the analysis Total Nitrogen (TN) and Dissolved Nitrogen (DN) in surface waters, ground waters, effluents and influents. The tests covered by this SOP are W-TN and W-DN.

1.2. Results are reported without qualification in the range from the practical quantitation limit (PQL) 0.050 mg N/L up to 0.50 mg N/L. Samples with higher concentrations are analyzed following SOP NU-106 or after dilution into the range. The method detection limit (MDL) is 0.020 mg N/L.

2. SUMMARY OF THE METHOD

2.1. The described procedure is based on ASTM D8083-16.

2.2. This method quantitatively measures nitrogen from organic and inorganic nitrogen compounds. The sample (300 ul) is combusted at 720 ºC in the presence of a platinum catalyst. Under these conditions all nitrogen containing compounds are converted to nitrogen oxide (NO). This is followed by a reaction with ozone to form excited state NO_2. As the NO_2 returns to the ground state, the emitted radiation is detected using a chemiluminescence detector.

2.3. This procedure is applicable to nitrogenous matter in the sample that can be injected into the apparatus reproducibly. The method does not measure N_2 as gas. An external sparge kit is used as a mixing mechanism to suspend small particles that may otherwise settle over time. The openings of the autosampler needle and injection syringe limit the maximum size of particles included in the analysis.

2.4. Interferences: Acid preservation of samples minimizes interferences that can cause results to be low. Gases of halide components may interfere with the detection system. They are eliminated by passing the reaction gas through a halogen scrubber. Samples with high and large particulate loading may cause TN analysis to be biased low (see 2.3.). Dissolved salts that are not volatized may result in devitrification or solidify in the catalyst tube resulting in decreased flow rates and decreased catalyst efficiency. Total organic carbon (TOC) up to 300 mg/L does not interfere.

3. EQUIPMENT AND SUPPLIES

3.1. TOC-L2-CPH : Shimadzu Total Organic Carbon Analyzer equipped with an autosampler, external sparge kit, and Total Nitrogen Module (TNM-L).

3.2. Assorted volumetric flasks (Class A)

3.3. Eppendorf and Oxford pipettes

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3.4. Fisher 24 mL Screw Thread Vials with PTFE/Silicone Septa (03-339-30H) or equivalent

4. CHEMICALS, 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 Reagent and Standard Logsheet for W-TN analysis. This excel sheet (NU-RGTSTD) is located in the REAGENT_PREP folder on the Nutrients drive located in the subfolder\\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\REAGENT_PREP\TOC\TN_TOC\Low level TN. It needs to be updated whenever a reagent or standard is newly prepared. For every analysis file print the Reagent and Standard Logsheet and place it into the analysis folder.

Note: Reagent and standard preparations must be documented in the Laboratory Information Management System (LIMS) 'Standard Preparation Tracker' module. Unique ID numbers for each standard and reagent are generated in the module and documented in the reagent and standard sheet submitted with each analysis folder. If available, a Certificate of Analysis for each purchased reagent and standard is maintained in the REAGENT_PREP folder on the Nutrients drive located in the subfolder \\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\REAGENT_PREP\TOC\TN_TOC\Certificates of Analysis. 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, please read their MSDS and the safety notes found in Section 11 of this SOP.

4.1. Deionized (DI) water

4.2. UHP Grade Compressed Air

4.3. Concentrated sulfuric acid (H_2SO_4) (A.C.S. Grade) and hydrochloric acid (HCl) (A.C.S. Grade)

4.4. Potassium Nitrate (KNO_3) (Certified) and Ammonium Sulfate ($(NH_4)_2SO_4$) (ACS Grade)

Stock Standard Solution (1000 mg N/L) - This solution is used for the preparation of the calibration standards. Holding time for the Stock standard is 180 days.

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4.4.1. Dry 8g of Potassium Nitrate and 5g of Ammonium Sulfate at 120ºC for 2 hrs. Cool in a desiccator.

4.4.2. Add approx. 800 mL of DI water to a clean 1 L flask. Add 2 mL of concentrated H_2SO_4 and mix.

4.4.3. Weigh out 3.609 g of the dried potassium nitrate into a weighing boat.

4.4.4. Weigh out 2.360 g of the dried ammonium sulfate into a separate weighing boat.

4.4.5. Rinse the contents of the weighing boats into the flask using DI water. Dilute to the mark and mix thoroughly. Label this flask 'TN STOCK 1000 PPM'.

4.5. KNO_3 Stock (50 mg N/L)- This solution is used for the preparation of the ICV/CCV, Spikes, and LFB. It is prepared from a different source or lot number as the KNO_3 used to prepare the calibration standards. Holding time is 90 days.

4.5.1. Dry 1g of Potassium Nitrate at 120 ºC for 2 hours. Cool in a desiccator.

4.5.2. Add approximately 800 mL of DI water to a clean 1 L flask. Add 2 mL of concentrated H_2SO_4 and mix.

4.5.3. Weigh out 0.3609 g of the dried potassium nitrate into a weighing boat.

4.5.4. Rinse the contents of the weighing boat into the flask using DI water. Dilute to the mark and mix thoroughly and transfer to a 1 L storage bottle. Label this flask '50 PPM KNO3'.

4.6. Preparation of QC-Standards - Holding time, 30 days.

4.6.1. Blank Standard/Diluent (0 mg N/L) - To a 2 L volumetric flask containing approx. 1.5 L of DI water, carefully add 4.0 mL of concentrated H_2SO_4. Dilute to the mark and mix thoroughly. Used for the method blank (MBLK) and continuing calibration blank (CCB). This solution is also used for W-TOC analysis (see SOP NU-062).

4.6.2. PQL and Calibration Standard (PQCK, 0.050 mg N/L) - To a 1 L volumetric flask containing approx. 900 mL of diluent (4.6.1.), add 0.050 mL of the TN STOCK 1000 PPM (4.4.), fill up to the mark with diluent and mix thoroughly. Transfer to a 1 L storage bottle and label as 'PQCK 0.050 PPM N'.

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4.6.3. Initial Calibration Verification/Continuing Calibration Verification Standard (ICV2/CCV2, 0.20 mg N/L) - To a 1 L volumetric flask containing approx. 800 mL of diluent (4.6.1.), add 4 mL of the 50 PPM KNO3 stock standard (4.5.), fill up to the mark with diluent and mix thoroughly. Transfer to a 1 L storage bottle and label as 'ICV/CCV/0.2 PPM N'.

4.6.4. 0.1 PPM N Calibration Standard (0.1 mg N/L) - To a 1 L volumetric flask containing approx. 900 mL of diluent (4.6.1.), add 0.100 mL of the TN STOCK 1000 PPM (4.4.), fill up to the mark with diluent and mix thoroughly. Transfer to a 1 L storage bottle and label as '0.1 PPM N'.

4.6.5. 0.2 PPM N Calibration Standard (0.2 mg N/L) - To a 1 L volumetric flask containing approx. 900 mL of diluent (4.6.1.), add 0.200 mL of the TN STOCK 1000 PPM (4.4.), fill up to the mark with diluent and mix thoroughly. Transfer to a 1 L storage bottle and label as '0.2 PPM N'.

4.6.6. 0.4 PPM N Calibration Standard (0.4 mg N/L) - To a 1 L volumetric flask containing approx. 900 mL of diluent (4.6.1.), add 0.400 mL of the TN STOCK 1000 PPM (4.4.), fill up to the mark with diluent and mix thoroughly. Transfer to a 1 L storage bottle and label as '0.4 PPM N'.

4.6.7. 0.5 PPM N Calibration Standard (0.5 mg N/L) - To a 1 L volumetric flask containing approx. 900 mL of diluent (4.6.1.), add 0.500 mL of the TN STOCK 1000 PPM (4.4.), fill up to the mark with diluent and mix thoroughly. Transfer to a 1 L storage bottle and label as '0.5 PPM N'.

4.7. Acid Rinse (ACID RINSE) for Catalyst Regeneration - To a 1 L volumetric flask containing approx. 800 mL of DI water, carefully add 164 mL of concentrated HCL. Dilute to the mark and mix thoroughly. This is also used for W-TOC analysis. Holding time, 90 days. (See NU-062)

4.8. ALTQC - A certified quality control standard is purchased. For the expiration date of the purchased stock refer to the manufacturer's certificate. The stock ALTQC standard may be diluted appropriately so that the concentration falls within the calibration range. Holding time of the diluted standard, 30 days.

4.9. Glycine ($C_2H_5NO_2$); Purity 99+%

Organic Nitrogen Stock Solution (1000 mg N/L)-This is used for the preparation of the Total Organic Nitrogen Check solution.

4.9.1. Dry about 3 g of glycine at 120^{o}C for 2 hours. Cool in a desiccator.

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4.9.2. Add approx. 150 mL of DI water and 0.5 mL H_2SO_4 to a clean 250 mL flask.

4.9.3. Weigh out 1.3392 g of the dried glycine into a weighing boat. Rinse the contents of the weighing boat into the flask using DI water. Dilute to the mark and mix thoroughly. Label this flask 'ORGANIC NITROGEN STOCK 1000 PPM'. Holding time, 180 days.

Note: This reagent (ORGANIC NITROGEN STOCK 1000 PPM) is also used for simultaneous TOC/TN analysis.

4.10. Total Organic Nitrogen Check (TONCHCK2, 0.2 mg N/L)-To a 250 mL volumetric flask containing approx. 100 mL of diluent (4.6.1), add 0.050 mL of the 1000 mg N/L glycine stock solution (4.9.). Dilute to mark and mix thoroughly. Label this flask 'TONCHCK2 0.2 PPM N'. Holding time, 30 days.

5. SAMPLE HANDLING AND PRESERVATION

5.1. Samples are preserved with sulfuric acid at collection and stored on ice or at ≤ 6 °C (not frozen). Samples submitted to the laboratory for DN analysis must be filtered in the field prior to acidification. All samples must be analyzed within 28 days of collection date for results to be reported unqualified.

5.2. Samples are stored in the Nutrient refrigerators in the receiving room of the FDEP laboratory. Allow samples to come to room temperature before processing.

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

6.1. Open program TOC-CONTROL-L.

6.2. Choose Sample Table Editor icon →hit ok.

6.3. To start a new file and run: Click on the *New* button in the top left corner. Select the H/W settings on the drop-down menu → TOC-L2-CPH-SPARGE. To save the file, click *Save* and name the file YYMMDDLN# (where # is a number 1, 2, etc., that indicates the 1st, 2nd, etc., run of the day. Ex.: 120211N1). → *Save*.

6.4. In some cases, it may be helpful to perform an optional rinse before the calibration. To add the rinse sequence to the beginning of the run (the rinse blank result will be calculated from a previous calibration), highlight line 1, right click, and select *Insert*→ *Multiple Samples*. This opens the Sample Group Wizard.

6.4.1. Page 1 Sample Source: Select *Calibration Curve* option and browse button. Then select calibration → *Open*; select folder icon → *Data* → *OK* → 'LOW LEVEL TN CAL CURVE. cal' → *Open* → *Next*.

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6.4.2. Page 2 Sample Parameters. Set options as follows:
Number of Samples = 1
Start Vial = 0
Sample Name = Enter file name (Ex.120211N1)
Sample ID = RINSE→ Finish

6.5. To setup a calibration sequence, highlight line two and then select *Insert* → *Calibration Curve* → Select folder icon → *Data* → *OK* → 'LOW LEVEL TN CAL CURVE.cal' → *Open*.

6.6. After inserting the calibration, select the autosampler image in top right corner. Assign vial numbers as follows:
0 mg N/L standard = 1 (diluent, 4.6.1.)
0.05 mg N/L standard = 2 (PQCK, 4.6.2.)
0.1 mg N/L standard = 3 (0.1 PPM N, 4.6.4.)
0.2 mg N/L standard = 4 (0.2 PPM N, 4.6.5.)
0.4 mg N/L standard = 5 (0.4 PPM N, 4.6.6.)
0.5 mg N/L standard = 6 (0.5 PPM N, 4.6.7.)
Select *OK* to complete. The vial column numbers should read 1,2,3,4,5,6.

6.7. To setup a sample sequence, highlight line three, select *Insert* → *Multiple Samples*. This will open the Sample Group Wizard.

6.7.1. Page 1 Sample Source; Select Calibration Curve option and browse button. Then select calibration file that was just created (6.5) and open → Next.

6.7.2. Page 2 Sample Parameters; Set options as follows:
Number of Samples = 87
Start Vial = 7
Sample Name = Enter file name.
Sample ID = Leave blank
→ Finish → OK.

6.8. Pour samples and Quality Control (QC)-standards into labeled 24 mL vials with septa according to the run sequence. Place them in the autosampler rack and return rack to turntable for realignment. Make sure that all vials are correctly seated.

6.8.1. For each batch: Select one sample from a batch of 20 or less samples as QC sample for the batch. Pour this sample in duplicate to assess precision.

6.8.2. Prepare a 0.20 mg N/L matrix spike (MS, spike code T1) by adding 80 µL of the 50 PPM KNO3 stock (4.5.) to one 20 mL portion of the selected

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sample. Prepare a laboratory fortified blank (LFB) following the same procedure as the 0.20 mg N/L matrix spike by substituting blank standard (4.6.1.) for the sample. Effluent samples are batched separately.

6.8.3. Each run begins with the following sequence:

Rinse (6.4) (optional)
Calibration (6.5)
ICV2/CCV2 (CCV, 0.2 mg N/L, 4.6.3.) ICV when calibration is new.
PQCK (PQL, 0.05 mg N/L, 4.6.2.)
ALT# (ALTQC, 4.8.)
TONCHCK2 (4.10.)
CCV2
CCB (Blank Standard, 4.6.1.)
These continuing calibration checks (CCV2 and CCB) are scheduled to **bracket every 10th analysis on the run**.
MBLKs (4.6.1.), **LFBs** (6.8.2.), sample duplicates (6.8.1.), and spiked samples (MS,6.8.2.)
Final **CCV2** and **CCB**

Note: The first CCV after a new calibration is called the Initial Calibration Verification standard (ICV).

6.9. Connect Instrument: Click *Connect.* Wait for connection and ready indicator, then press *Start*→ Check Shut down instrument box → press Run to start the run.

Note: Print the run file that was just created by going to *file* → *print*→ *sample table*.

6.10. Post-run Procedure: Results are exported as an ASCII file and converted into a raw file in the QC manager for performing calculations and uploading to LIMS.

6.10.1. *File* → *ASCII Export Setting* → *Detail* → *Data.* Check the following export options; Sample ID, Dilution, Date/Time, Mean Conc., Result, and Vial. Also make sure the export data box is checked. Under the "Misc" tab that asks for the separator settings, choose "tabulator," and check the box for "Export strings in quotation marks…".

6.10.2. To export file: *File* → *ASCII Export* → *Detail* (Include Injection Data) → *Filename* → *Save.*

6.10.3. Export to C:\TOC-L\Data for TOC-L2. This is the local drive for the instrument workstation.

6.10.4. Open C:\TOC-L in Windows Explorer. Locate the text file you just

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created, as well as the peak (*.pkt), calibration (*.cal) and sample table (*.t32 or *.tlx) files associated with the analysis and copy all four files from the local drive to the appropriate instrument folder (TOC-L2) in the current year's subfolder located at \\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\Tocv\TOC-L2_TN
Note: the exact location of the data files within instrument folder structure is determined by the software settings (Tools > Organize Files; please see User manuals for details).

6.10.5. Print a copy of the final report and data table containing injection peaks by selecting: *File* → *Print* → *Sample Report –All*, then *File* → *Print* → *Sample Table.*

6.11. Quantitation/Calculations:

6.11.1. The instrument will give the values for nitrogen in mg N/L. The value given by the instrument is the average of three injections for a given sample that have a percent relative standard deviation of less than 5 %. The calibration consists of five standards plus a blank. The regression model applied is linear.

6.11.2. The QC Manager will calculate spike and standard recoveries and RPDs. For general calculations including RPDs and spike recoveries, see the FDEP Quality Manual Section 5.9.

6.11.3. The calibrants are all prepared manually (see section 4.5. and 6.6.). The correlation coefficient for the calibration is calculated by the instrument software. Use the NU-TN form located at \\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\Tocv\ to calculate the recoveries of the calibrants. Make sure you use the latest version of the form. Open the form, enter the header information, the slope, intercept, and mean area results to calculate the recovery of calibrants. Acceptance limits for the recoveries of the calibration standards above the PQL are 90 – 110 % and 70 – 130 % for the PQL-level standard. The acceptance limits for blank calibration point results are below the MDL and above the negative value of the MDL (-MDL < blank < MDL).

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-TN), filename/s (YYMMDDN#), and initials of the analyst.

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7.2. Each report folder should contain the following items - run report including raw data and calibration curve, calibration standard recovery sheet, results report method parameters, bench sheet, Reagent and Standard Logsheet, and a copy of the data and QC assessments uploaded from QC Manager to LIMS at the time of test authorization.

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

7.4. Instrument raw data files, including peak (*.pkt), calibration (*.cal) and sample table (*.t32 or *.tlx) files, are backed up from instrument PCs and stored on the Nutrients drive under \\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\Tocv\TOC-L2_TN

7.5. TN instrument data text transfer files for upload to QC manager are stored in electronic format in appropriate instrument folder (TOC-L2) in the current year's subfolder located at \\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\Tocv\TOC-L2_TN

7.6. Method validation data is on file with the supervisor. Digital copy is also stored on the network, under an appropriate sub-folder in \\fldep1\dear\Labs\Lab5\Chemistry\Inorganic\Nutrient\Validations.

8. MAINTENANCE/TROUBLESHOOTING

Note: Make sure all maintenance performed is documented in the instrument's maintenance log.

8.1. Maintenance:

8.1.1. Before beginning sample analysis, conduct a general inspection of the instrument to ensure that everything is in order, and that there is sufficient UHP Air to complete the run.

8.1.2. The humidifier and dehumidifier vessels should be filled with DI water to the fill line on the vessel. Once a month, the humidifier vessel should be drained and refilled with fresh DI water. This prevents contamination.

8.1.3. Keep the rinse water bottle filled with DI water. If running samples overnight, fill the bottle with DI water before starting the run.

8.1.4. The diluent bottle should be changed with fresh Blank Standard/Diluent (4.6.1.) at least once per month.

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8.1.5. Make sure the HCL Intake is changed with fresh 2N HCL (4.7.) at least once every three months.

8.1.6. Inspect the slider and O-rings before analysis and clean with DI water if needed.

8.1.7. Check the waste line daily to ensure proper flow. Waste backup will damage the autosampler. Dump waste down the drain as needed.

8.1.8. Regeneration of TC catalyst should be performed on a case by case basis TC catalyst regeneration has little effect on TN analysis. See instrument manual for regeneration procedure. Regeneration should be followed with a run of at least 20 CCBs and a few CCVs.

8.1.9. Other maintenance may be required periodically, such as replacing catalyst, replacing halogen-scrubber, maintaining syringe and 8-port valve. Please see User's manuals for details.

8.2. Troubleshooting Notes:

8.2.1. To stop the run at any point and delete the calibration,
Click *Stop* button → *Stop* (stop all processes immediately).

8.2.2. To interrupt the run at any point and save the calibration,
Click *Stop* button → *Stop* (after current sample is completed).

8.2.3. In order to restart a run in progress (for example, if there is a QC failure at the beginning of a run) and keep the sample table set up for a new run, previous data gathered or attempted must be deleted prior to restart. Highlight the lines to delete, go to *Edit* → *Clear Measured Data* → *Highlighted* → *Yes*, then click *View Vial Settings* → Assign the vial numbers for rerun samples.

8.2.4. Autosampler lid must be in place for run to proceed properly.

8.2.5. Treat samples suspected to contain humic acids as follows:
a. Split a well-mixed sample into two portions.

b. Keep one portion at pH $\leq$ 2 and adjust the other to pH 5-7 with 10 N Sodium Hydroxide to increase the solubility of humic acid.

c. Cap and allow both portions to sit at room temperature for ½ hour.

d. Analyze both splits and report the highest result.

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

9.1. A new calibration must be performed at the beginning of the analysis and the linear calibration range (LCR) must be verified (see 9.2.). Consecutive runs on one day may use the same calibration. The linear calibration consists of five standards (0.5 mg N/L, 0.4 mg N/L, 0.2 mg N/L, 0.1 mg N/L, and 0.05 mg N/L) and a blank. It is followed by a sequence of initial quality control standards as described in section 6.8.

9.2. LCR: See the FDEP Quality Manual Appendix B, Section 7.0, 'Protocol for Determining the Test Method Range of Applicability'. The correlation coefficient of the linear regression (r^2) must be equal to or larger than 0.995. Acceptance limits for the recoveries of the calibration standards above the PQL are 90 – 110 % and 70 – 130 % for the PQL-level standard. The acceptance limits for blank calibration point results are below the MDL and above the negative value of the MDL (-MDL < blank < MDL).

9.3. Each analytical run may contain a number of sample batches, with no more than 20 samples per batch. Each batch is required to have a MBLK, an LFB, a matrix spike, and a duplicate sample. The spikes are prepared according to section 6.8.2. Acceptance limits for the LFB and matrix spike recoveries are 85-115 %.

9.4. On every analytical run, one organic nitrogen check (TONCHCK2) is run at the beginning of the run (4.10.). The result must be between 85-115 % recovery. If the recovery is outside the control limit the samples must be reanalyzed or reported qualified. The instrument should be checked to ensure that it is operating correctly.

9.5. The AltQC standard (ALT#, 4.7.) is a second source certified standard with acceptance limits supplied by the manufacturer (Independent Reference Material, IRM). One AltQC standard is analyzed with every analytical run.

Note: *Currently there are no certified reference standards available for TN. The 'Simple Nutrients' standard was analyzed adding the certified values for ammonia and nitrate/nitrite to reflect TN. The acceptance limit for the sum was taken from the more stringent limit of the individual components.*

9.6. One PQL (PQCK, 4.6.2.) level standard is analyzed for every analytical run. For analytical runs that are longer than 24 hours, at least one PQL standard is analyzed every 24 hrs. Acceptance limits for the PQCK recovery are 70-130 %. The PQCK and MBLK analysis results are uploaded to LIMS whenever samples are reported and used for ongoing annual MDL and PQL verification.

9.7. A CCV2/ICV2 (CCV, 4.6.3.) and a blank (CCB, 4.6.1.) standard are run at the

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beginning of every run, after every 10th analysis, and at the end of the run. Acceptance limits for the recoveries of the check standard are 90-110 %.

9.8. The sample/matrix spike precision should be less than 20 % RPD for results over the practical quantitation limit (PQL).

9.9. Acceptance limits for the blanks (MBLKs and CCBs) are < 0.020 and > -0.020 mg N/L.

9.10. For general 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 SOP NU-043. If there are any anomalous results (e.g., differing results for multiple analyses of same sample) or any unusual events (spilled sample, cannot locate sample, etc.), see supervisor for further instruction.

10. SAFETY/WASTE MANAGEMENT

10.1. Refer to the MSDS notebook for information on safe handling of samples and reagents used with instrument.

10.2. Flush sample waste (as long as no other known hazards are present) down the sink with plenty of water.

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

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

11. 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.

11.1. ASTM D8083-16.

11.2 Shimadzu TOC-L/TOC-L2 Manual.

11.3. Quality Manual, Bureau of Laboratories, FDEP.

11.4. DEP SOP NU-062, Determination of Total Organic Carbon (TOC) and dissolved Organic Carbon (DOC) Using the Shimadzu TOC Analyzer

11.5. DEP SOP NU-033, Summary of Analysis Procedures for the General Chemistry

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and Nutrients Workgroups.

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

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

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

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

11.10. Laboratory Health and Safety Plan.

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

APPENDIX OF SIGNIFICANT CHANGES

01/29/2020 SECTION 4: Added clarification that the ICV/CCV stock solution is prepared from a different source or lot of KNO_3 as used for the calibration standards.

02/14/2020 SECTION 6 AND SECTION 9: Clarified the acceptance limits for the blank calibration point in the calibration curve.

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