DETERMINATION OF NITRITE IN WATER USING THE AQ2 DISCRETE ANALYZER

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1. SCOPE AND APPLICATION

1.1. This SOP is applicable to the determination of nitrite in surface, ground and saline water, and in domestic and industrial wastes. The applicable range for this method is from the method detection limit (MDL) of 0.002 mg N/L to 0.20 mg N/L. Samples containing levels higher than 0.20 mg N/L should be diluted to be within the calibration range.

Key Parameters:

Calibration range	0 – 0.20 mg N/L
Minimum Detection Limit (MDL)	0.002 mg N/L
Practical Quantitation Limit (PQL)	0.005 mg N/L

1.2. Tests covered by this SOP are W-NO_2 and W-NO_2-F.

2. SUMMARY OF THE METHOD

2.1. This method is based upon EPA method 353.2, Rev. 2.0 (1993) and Seal Analytical AQ2 method EPA-137-A Rev.1.

2.2. The diazonium compound, formed by diazotation of sulfanilamide by nitrite in water under acid conditions, is coupled with N-(1-naphthyl)-ethylenediamine dihydrochloride (NED) to produce a reddish-purple azo dye, which is measured colorimetrically at a wavelength of 520 nm.

2.3. Interferences: Background absorbance (color) at the analytical wavelength may interfere. Residual chlorine in the sample may cause low spike recoveries – this can be removed by adding sodium thiosulfate to the sample aliquot prior to spiking (see section 5.4 in DEP SOP NU-066 for chlorine test and treatment procedures). Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that bias analyte response.

3. EQUIPMENT AND SUPPLIES

3.1. Instrumentation

Instrumentation Unique ID	Component	Manufacturer	Model(s)/Catalog #
AQ2-2	Automated Discrete Analyzer	Seal	AQ2 (S/N:090656)

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3.2. Instrument Accessories

Accessory	Manufacturer/Vendor	Model(s)/Catalog/Part Number
Lamp Assembly	Seal	5100
Aspiration Probe	Seal	5110
10 mm Cuvette Inlet Tubing	Seal	5671
10 mm Cuvette Outlet Tubing	Seal	5672
Peristaltic Pump Tubing	Seal	5140
Syringe Body, Piston, O'Ring	Seal	5195-1
Reagent Compartment -Tray	Seal	P/N: MA000106S
Sampling Station -Probe	Seal	P/N: 5125-S
Sample Tray (57 Position Tray)	Seal	P/N: 5350
Reaction Ring – Segment	Seal	P/N: 5000

3.3. Equipment

Equipment	Manufacturer/ Vendor	Model(s)/Catalog Number
Analytical Balance	Mettler Toledo/Fisher	MS204TS
Stirrer	Fisher Scientific	11-100-49S (Discontinued)
Stir Bars, Assorted	Fisherbrand/Fisher	14-513-61; 14-513-58; S68069
Adjustable Pipette, 10-100 µL	Eppendorf/Fisher	3123000047/13690029
Adjustable Pipette, 100 -1000 µL	Eppendorf/Fisher	3123000063/13690032
Adjustable Pipette, 1-10 mL	Eppendorf/Fisher	3123000080/13690034
Filtering Flask, 1 L	Pyrex/Fisher (SR)	10180F
Glass Beaker, Assorted	Pyrex/Fisher (SR)	02-540-K; 02-540L; 02-540P;
Volumetric Flask, Class- A, Assorted	Pyrex/Fisher (SR)	10-210-5C; 10-210-5E; 10-210-5F; 10-210-5G; 10-210-5H
Magnetic Filter Funnel, 47 mm	Andwin Scientific/Fisher	NC0006472
Graduated Cylinder, assorted	KIMAX/Fisher (SR)	08-548D; 08-564A

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Equipment	Manufacturer/ Vendor	Model(s)/Catalog Number
Glass Funnel	Fisher (SR)	10-346-5B
Or Equivalent		
Note: (SR) designates supply is typically ordered through the in-house stockroom		

3.4. Consumables and Supplies

Supplies	Manufacturer/ Vendor	Model(s)/Catalog Number
Reaction Segment	Seal	P/N: 5000
Reaction Container	Seal	P/N: 5010
2 mL Sample Cup	Seal	P/N: 6015
Sample Beakers, Polypropylene, 20 mL	Fisher (SR)	08-732-120
Sample Beakers, Polypropylene, 50 mL	Fisher (SR)	08-732-122
Pipette Tips, 200 µL	Eppendorf/Fisher (SR)	05-403-41
Pipette Tips, 1000 µL	Eppendorf/Fisher (SR)	05-403-43
Pipette Tips, 1-10 mL	Eppendorf/Fisher (SR)	05-403-116
Tube Centrifuge 15 mL Polypropylene	Corning/Fisher	05-538-59A
Weigh Dish, Hexagonal 1 ½ X 1	Fisher (SR)	02-202-100
Weigh Dish, Hexagonal 4 X 3	Fisher (SR)	02-202-102
Weigh Dish, Aluminum, 110 mm	Fisher (SR)	08-732-100
Transfer Pipet, 5 mL	Fisher (SR)	13-711-5AM
Parafilm	Fisher (SR)	13-374-12
Nitrocellulose Membrane	Fisherbrand/Fisher	SA1J792H5
Or Equivalent		
Note: (SR) designates supply is typically ordered through the in-house stockroom		

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

Note: Prior to using the chemicals in this analysis, please read their MSDS and the safety notes found in the safety/hazardous waste section of this SOP.

Note: All 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 Logsheet (NU-RGTSTD). This excel sheet needs to be updated and submitted as part of the analysis report folder. If available, a Certificate of Analysis for each purchased standard or reagent is maintained in the \\fldep1\dear\Labs\Lab3\Chem_Data\Inorganic\Nutrients\REAGENT_PREP\NO2 AQ2 folder. 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

Chemical	Alias	Purchased From	Catalog Number	Manufacturer	Concentration/ Purity	Expiration	Storage Temperature
Nitrite Waste WatR	SIM NUT AMP	ERA	770	ERA	varied	See CoA	See CoA
Sodium Hydroxide	NaOH	Fisher (SR)	S318-3	Fisher Chemical	ACS, ≥ 97%	see CoA	Room
Ethylenediamine Tetraacetic Acid	EDTA	Fisher	S311-500	Fisher	Crystalline/Certified ACS	None	Room
Ammonium Chloride	NH_4Cl	Fisher	A661-500	Fisher Chemical	ACS, ≥99.5%	see CoA	Room
N-(1-Naphthy)-Ethylenediamine	NED	Fisher	AC42399-0250	Acros Organics	ACS Reagent	None	Room
o-Phosphoric Acid	H_3PO_4	Fisher (SR)	A242-212	Fisher	85% Certified ACS	None	Room
Sodium Chloride	NACl/ Salt	Fisher (SR)	S271-500	Fisher	Crystalline/ Certified ACS	None	Room
Sulfnilamide	$C_6H_8N_2O_2S$	Fisher (SR)	AC132855000	Acros Organics	0.98%	None	Room
Sulfuric Acid	H_2SO_4	Fisher (SR)	A300S-212	Fisher	Certified ACS Plus	None	Room
Chloroform	$CHCl_3$	Fisher	C298-500	Fisher	Certified ACS	None	Room
Potassium Nitrite	KNO_2	Fisher	P267-500	Fisher	Certified ACS	None	Room

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Chemical	Alias	Purchased From	Catalog Number	Manufacturer	Concentration/ Purity	Expiration	Storage Temperature
Bleach, Generic	NaOCL For Cleaning	Store Brand	N/A	No Clorox	No additives, ~6%	N/A	Fridge
Deionized Water	H_2O	In-House	N/A	In-House	N/A	None	Room
Or Equivalent							
Note: (SR) designates supply is typically ordered through the in-house stockroom.							
Note: Routinely used expiration ranges are listed in the table. Follow CoA if more stringent.							

4.2. Reagents

4.2.1. NED Color Reagent - To a 600 mL beaker containing about 250 mL of deionized (DI) water add 2.0 g sodium hydroxide pellets and 1.0 g ethylenediamine tetraacetic acid disodium salt hydrate (EDTA). Stir to dissolve completely. Add 50 mL of concentrated phosphoric acid. Caution, the beaker will become warm! While stirring add 50 g ammonium chloride, 12.5 g sulfanilamide and additional DI water to bring the volume to about 450 mL. Dissolve completely. Add 0.625 g N-(1-naphthyl)-ethylenediamine dihydrochloride (NED) and stir to dissolve completely. Dilute to 500 mL. Filter the solution using a 0.45 micron filter and transfer to a dark container. Store in the refrigerator. Holding time, 30 days. Discard if it develops a significant pink color.

4.2.2. Alkaline EDTA Rinse (Cuvette Wash) - Add approximately 250 mL DI water to a 500 mL volumetric flask. Add 5 g sodium EDTA (4.1.) and 10 g NaOH (4.1.) to the flask and swirl until the NaOH dissolves. Dilute to volume with DI water and mix well. Holding time, 60 days.

4.3. Standards

4.3.1. QCNI, a second source QC standard, should be prepared from a stock from a different manufacturer or a different lot than the calibration standards. A certified standard, such as the ERA SIM NUT (4.1), is preferred. A certified standard should be diluted with DI water if needed to bring the concentration of the stock standard approximately into the middle of the calibration range.
4.3.1.1. For the ERA standard (ERA SIM NUT (4.1)), follow the manufacturer instructions to prepare the certified concentration standard from the ampule. Name the standard to indicate it's source, e.g. "NITRITE_ERA_P297-770." Store refrigerated. Holding time as recommended in the CoA or 180 days.
4.3.1.2. If the concentration of the certified standard (4.3.1.1.) is near the mid-calibration range (0.10 mg N/L) or below, name the standard QCNI and use it on the run. If the concentration of the certified standard is too

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high, dilute it with DI water to prepare the QCNI standard. Into a 1 L flask containing approximately 500 mL DI water, pipette appropriate volume (**V_1**) of the certified standard based on the formula below.

$$M_1V_1 = M_2V_2,$$

where M_1 = Concentration of the certified standard - concentration can be obtained from the Certificate of Analysis
V_1 = Volume of stock required
M_2 = Concentration of working standard
V_2 = Volume being prepared = 1.0 L

Bring to volume with DI water and invert 12 times. Store in refrigerator. Holding time, 30 days.

4.3.2. Stock Nitrite Standard, 2000 mg N/L - Pour the Potassium Nitrite (KNO_2) in a glass beaker and place it in an oven for 24 hours at 105 °C. Dissolve 12.14 g of the dried Potassium Nitrite in approximately 800 mL of DI water contained in a 1 L volumetric flask. Add 2 mL of chloroform. Bring the volume to 1 L with DI water, invert 12 times and refrigerate. Holding time, 180 days.

4.3.3. Working Stock Standard, 20.0 mg N/L - Into a 1 L flask containing approximately 500 mL DI water, pipette 10.0 mL of the 2000 mg N/L nitrite stock standard (4.3.2.). Bring to volume with DI water and invert 12 times. Store in refrigerator. Holding time, 90 days.

4.3.4. Working Stock Standard, 0.20 mg N/L - Add 2.0 mL of the 20.0 mg N/L working stock standard (4.3.3.) to a 200 mL volumetric flask containing approximately 150 mL of DI water. Bring to volume with DI water and invert 12 times. Store in refrigerator. Holding time, 30 days.

4.3.5. Continuous calibration verification standard, CCV, 0.10 mg N/L - Add 5 mL of the 20.0 mg N/L working stock standard (4.3.3.) to a 1 L volumetric flask containing approximately 800 mL of DI water. Bring to volume with DI water and invert 12 times. Store in refrigerator. Holding time, 30 days.

4.3.6. PQNI (PQL level standard), 0.005 mg N/L – Add 50 µL of the 20 mg N/L working stock standard (4.3.3.) to a 200 mL volumetric flask containing approximately 150 mL of DI water. Bring to volume with DI water and invert 12 times. Store in refrigerator. Holding time, 30 days.

4.3.7. Working Nitrite Spike Standard, 4.0 mg N/L - Add 500 µL of the 2000 mg

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N/L nitrite standard (4.3.2.) to a 250 mL volumetric flask containing approx. 100 mL of DI water. Dilute to volume, invert 12 times and refrigerate. Holding time, 90 days.

5. SAMPLE HANDLING AND PRESERVATION

5.1. Samples are collected and stored on ice or in refrigerator at ≤ 6°C (not frozen).

5.2. Samples must be analyzed within 48 hours of collection time.

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

Note: Samples containing particulate matter should be filtered through a 0.45 µm filter prior to analysis on the AQ2 Discrete Analyzer.

Note: 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 standards and reagents from the refrigerator and check the expiration dates. Discard expired standards and/or reagents and reprepare if necessary.
6.1.2. Use 57-position sample tray for the run.

6.2. Calibration:

6.2.1. The nitrite calibration is prepared from the 0.20 mg N/L working stock standard (4.3.4.) by autodilution on the instrument.

Concentration (mg N/L)	Source
S1 - 0.0000	DI H_2O – Pour in cup
S94 – 0.2000	0.20 mg N/L working Stock Standard (4.3.4) - Pour in cup
S90 – 0.0048	Auto Dilution
S91 – 0.0100	Auto Dilution
S92 – 0.0400	Auto Dilution
S93 – 0.1000	Auto Dilution
S0 = 0.0000	DI H_2O – Pour in cup

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6.2.2. The diluent for nitrite analysis is DI water.

6.3. Reagents, Run Controls, and Stock Standard Loading:

6.3.1. Label one AQ2 reagent vessel and fill it with the NED color reagent (4.2.1.).

6.3.2. Reagent Location - The reagent tray is located on the right side of the injector arm. It holds all reagents and controls (CCV and blank) in the assigned locations. Place the NED color reagent (4.2.1.) in position 7.

6.3.3. Controls (CCV/check and CCB/blank) – The 0.1 mg N/L standard is used as the CCV standard (4.3.5.). The instrument blanks or CCBs are DI water. CCV and CCB standards should be run every 10 samples and at the beginning and end of each run (8.2.) Previous experience shows that pouring CCV and CCB into AQ2 reagent vessels may result in random failures. It is recommended, therefore, to pour CCV and CCB standards into 2 mL sample cups and place them in the 57-position sample tray based on the run sequence.

6.4. Batch QC:

6.4.1. A laboratory fortified blank (LFB) and a sample either ran in duplicate and/or spiked in duplicate must be analyzed for every batch of 10 samples. Use the 4.0 mg N/L stock solution (4.3.7.) as the spiking solution. Add 150 µL to 10 mL of sample (or DI water in the case of an LFB) for a 0.060 mg N/L spike. The spike code in QC Manager is LO.

6.4.2. One method blank (MBLK) must be analyzed for every batch of 10 samples. DI water is used for the MBLK.

6.5. Analysis Setup/Sequence Setup:

6.5.1. Turn on the computer and start the software by double-clicking on the AQ2 icon on the screen. The username is '**Instrument 1, 2 or 3' depending on the instrument used** and the password is 'password'. Next turn on the AQ2 Analyzer (there are two switches, turn on the large switch on the right side behind the instrument - the small switch right next to it is for the chiller and is not used for this test).

6.5.2. Clean and refill the DI water reservoir and the waste containers. Replace all used reaction segments.

6.5.3. In the software: Go to the 'Settings' menu and select maintenance. Zero the reaction segment counter.

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6.5.4. Go back to the 'settings' menu and select the daily start-up option.

6.5.5. Setup of the analytical run: Click on the scheduling icon on the left side of the screen. Select a free tray and **reagent set 2**. Name the run as follows:

Instrument 1: NOyymmddK1(use 2 for the 2nd run that day, etc.)
Instrument 2: NOyymmddQ(1)
Instrument 3: NOyymmddR(1)

Now the sequence screen appears. Leave lines one and two empty. Type the sample sequence in the 'Sample ID' column. Highlight the typed lines and select the NO_2 test. The test should appear in the requested test column to the right of the sample ID's. Click the 'save' icon. Click on the 'print' icon to print the bench sheet.

An example run sequence (see Section 9 for use of the Alkaline EDTA rinse (4.2.2) in case of carryover) includes:

- **Calibration**
- **CCV** (4.3.5.)
- **CCB** (6.3.3.) DI water
- **MBLK** (6.4.2.) DI water
- **PQNI** (4.3.6.)
- **QCNI** (ALTQC, 4.3.1.)
- **LFB** (6.4.1.)
- **Batched samples**, with duplicate spikes (6.4.1.) for each batch
- **CCV**
- **CCB**

6.5.6. Pouring the analytical run - Cup positions #1 and #2 are reserved for the calibration standards. For NO_2 the calibration standard (4.3.4.) is placed in position #2 (position #1 on the sample tray is unused). Now, start pouring the samples according to the cup position on the bench sheet.

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

6.5.8. 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 6.5.7. While the run is in progress, the method should be printed to be included in the analysis file. Go to the 'settings' menu and select the option 'Test Parameters'. Select the Test 'EPA353_2 Inst1, 2 or

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3' and print.

6.5.9. If the run quality deteriorates (unacceptable calibration and/or QC results, high blanks, sample carry-over, etc.), the system should be washed with the alkaline EDTA rinse solution (4.2.2.) to remove any reagent deposits in the aspiration tube and cuvette. If carryover is expected or observed during a run, the Alkaline EDTA Rinse (4.2.2.) can be scheduled after every sample cup in the method parameters. The solution is placed in a reagent vessel in position 1 of the reagent tray and is called Control 1 in the sample sequence.

6.6. Post-Run Procedure:

6.6.1. Review water baseline values ("W" column) before accepting the results. Anomalous readings here may be indicative of the instrument performance problems. Any results associated with such readings should be questioned.

6.6.2. After the run has finished click on the 'Reporting' icon on the left side of the screen. Select the appropriate reporting format and print the run report in the chronological reporting format. It contains the calibration and all the data.

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

6.6.4. 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 convention described in 6.5.5. 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 at \\FLDEP1\DEAR\Labs\lab3\Chem_Data\Inorganic\Nutrients\AQ2\Nitrite .

6.6.5. Convert the excel file into Raw data in the QC Manager. The Instruments used are 'Nutrients: Seal AQ2 1', 'Nutrients: Seal AQ2 2' or 'GenChem: Seal AQ2 3'.

6.7. Quantitation/Calculations:

6.7.1. The instrument will give the analysis results for Nitrite in mg N/L.

6.7.2. The QC Manager calculates relative percent differences (RPDs) for duplicate samples as well as recoveries for QC standards. For general calculations including RPDs, see Section 5.9 of the FDEP Quality Manual. Once calculated the results are uploaded into LIMS.

6.7.3. The high calibration standard is prepared according to section 4.3.4. The other standards used in the linear calibration are auto-diluted by the

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instrument from the high standard. The calibration consists of five standards and a calibration blank. The correlation coefficient for the calibration and the % error of the standards are calculated by the instrument software.

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(s) (W-NO2, W-NO2-F), filename/s (NOYYMMDDQ# (where Q is for AQ2 2 (see section 6.5.5. for other AQ2 instrument codes), and the # indicates the 1st, 2nd etc. run of the day), and the initials of the analyst. If nitrate by calculation was performed as well, the test W-NO3 is added to the label.

7.2. Each report folder should contain the following items - the run report(s), bench sheet(s), reagents and standard logsheet, the backlog containing the batching documentation, and a copy of the data and QC assessments uploaded from QC Manager to LIMS at the time of test authorization. If nitrate by calculation was performed a copy of the nitrate by calculation excel sheet (see DEP SOP NU-066, section 9) should be included. If sample dilutions were prepared, the dilutions recipes and the pipettes used are documented on the NU-PIPETTE form.

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

7.5. 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. QUALITY CONTROL/DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES (SEE DEP SOPS 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 the following DEP SOPs: NU-038 and NU-043.

8.2. The 0.1 mg N/L CCV standard (4.3.1.) and CCB will be run every 10 samples and at the beginning and end of each run. Acceptance limits for the recovery of the CCV are 90-110 %. Acceptance limits for the MBLK and MDL are < 0.002 and > - 0.002 mg N/L. For convenience, the QCNI standard (8.3) may be used as the CCV.

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8.3. The 0.1 mg N/L QCNI standard (4.3.1.) is prepared from a different source than the calibration standards. It serves as the ICV (initial calibration verification) standard and must be run after the calibration standards to verify the calibration. Acceptance limits for the recovery of the QCNI are 90-110 %.

8.4. The PQL level standard (PQNI, 4.3.6.) is run on every analytical run and serves to verify the instrument performance at the low concentration range and as the ongoing MDL and PQL verification spike. Acceptance limits for the recovery of the PQNI are 70-130 %.

8.5. An LFB, a matrix spike and a sample or matrix spike duplicate are analyzed for every batch of 10 samples. Acceptance limits for the recovery of the LFB and spikes are 90-110 %. The sample/matrix spike reproducibility should be less than 20 % RPD for samples above the PQL.

8.6. Linear Calibration Range (LCR): See FDEP Quality Manual Appendix B, Section 7.0., 'Protocol for Determining the Test Method Range of Applicability'. The correlation coefficient of the linear calibration must be equal to or greater than 0.995. Acceptance limits for the percent error of calibration standards above the PQL are ±10%. Acceptance limits for the percent error of the PQL level standard (0.048 mg N/L) are ±30%. The acceptance limits for the blank calibration point results are below the MDL and above the negative value of the MDL (-MDL < blank < MDL).

8.7. Every day samples are analyzed a new calibration is performed at the beginning of the analysis and the LCR must be verified. Consecutive runs on one day may use the same calibration.

9. SAFETY/HAZARDOUS WASTE MANAGEMENT/POLLUTION PREVENTION

10.1. Review the Laboratory Health and Safety Plan available on the Laboratory Internet site. Choose the 'Operations' menu and select 'Health and Safety'.

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

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

10.4. Instrument and sample waste are flushed down the drain with plenty of water.

10. REFERENCES/DEFINITIONS

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.

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11.1. EPA method 353.2, Revision 2.0, August 1993.

11.2. Seal Analytical AQ2 method EPA-137-A Rev.1.

11.3. AQ2 Operator Handbook and AQ Software guide.

11.4 Quality Manual, Bureau of Laboratories, DEP.

11.5. Laboratory Health and Safety Plan.

11.6. DEP SOP NU-033, Summary of Sample Analysis Procedures for the General Chemistry and Nutrients Workgroups.

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

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

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

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

11.11. DEP SOP NU-066, Determination of Nitrate and Nitrite in Water Using the Lachat QuickChem 8500 Autoanalyzer

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

Appendix of Significant Changes

12/01/2010 Changed MDL level to 0.002 mg N/L and PQL to 0.005 mg N/L. Made changes to reflect the use of all three Seal AQ2 instruments.

11/29/2011 A section was added to list the chemicals used for the Nitrite analysis.

11/19/2012 A troubleshooting section was added to help restore/maintain run quality using an alkaline/EDTA wash procedure.

11/05/2015 Data archival information was clarified in Section 7.

12/27/2018 Section 4 is modified to include a reference to new storage location for certificates of analysis. In Section 8, added a reference to use of the PQL level standard for the ongoing MDL and PQL verification data collection. Section 10

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waste disposal procedure is clarified.

02/13/2020 Added control limits for the blank calibration standard.

11/25/2020 Added clarifications to section 8 on use of the ICV.

03/24/2021 Clarified Section 1. Revised Sections 3, 4, and 6 to add information tables. Revised 4.3.1. to include preparation of QCNI for the ERA certified standards.

12/19/2021. Revised section 4.3.1. and added 4.3.5. for new procedure of preparation of the QCNI and CCV standards.

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