DETERMINATION OF SULFATE, CHLORIDE AND NITRATE USING THE DIONEX ION CHROMATOGRAPHY SYSTEM

TABLE OF CONTENTS

Page

1. SCOPE AND APPLICATION 2
2. SUMMARY OF THE METHOD 2
3. EQUIPMENT AND SUPPLIES 2
4. REAGENTS AND STANDARDS 3
5. SAMPLE PREPARATION PROCEDURE 5
6. SAMPLE ANALYSIS/QUANTITATION 6
7. POST-RUN PROCEDURE 10
8. QUALITY CONTROL/DATA ASSESSMENT 11
9. DATA ARCHIVAL 12
10. MAINTENANCE 13
11. SAFETY/HAZARDOUS WASTE MANAGEMENT/POLLUTION PREVENTION 14
12. REFERENCES/DEFINITIONS/VALIDATION DATA 14

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1. SCOPE AND APPLICATION

1.1. This method details the determination of sulfate, chloride, and nitrate, in aqueous matrices (drinking, fresh and saline surface waters, effluents, influents) and solid matrices (soils, sediments). The calibration range is from the method detection limit (MDL) 0.20 to 120 mg Cl/L for chloride, 0.20 to 80 mg SO_4^{2-}/L for sulfate, 0.050 to 10 mg N/L for nitrate. The range may be extended by dilution. The sulfate MDL for solid matrices is 6.0 mg SO_4^{2-}/kg.

1.2. The aqueous tests covered are W-SO4-IC(-F), W-CL-IC(-F), and W-NO3-IC(-F)

1.3. For solid matrices, an aqueous extraction is performed (see DEP SOP NU-044) and the resulting extracts are analyzed. The solid tests covered by this SOP are S-SO4-IC, S-CL-IC, and S-NO3-IC.

2. SUMMARY OF THE METHOD

2.1. This method is based on EPA method 300.0, Rev. 2.1 (1993).

2.2. Anions are injected into the ion chromatograph and separated on an AS 18-4 µm 2x150 mm analytical column. A potassium hydroxide gradient solution serves as the mobile phase.

2.3. Once separated, the anions pass through an anion suppressor where they are converted to their more conductive acid forms and the potassium hydroxide eluent is converted to less conductive water. The separated anions in their acid forms are measured by conductivity and are identified on the basis of retention times as compared to standards.

2.4. Ion chromatography eliminates the need to use hazardous reagents and it effectively distinguishes among the halides (Br^-, Cl^-, and F^-) and the oxy-anions (SO_3^-, SO_4^-, NO_2^-, and NO_3^-).

2.5. For a description of possible interferences, see 300.0, Rev. 2.1 (1993).

3. EQUIPMENT AND SUPPLIES

3.1. Dionex Ion Chromatography System:

3.1.1. Dionex IC-5000+

3.1.2. Dionex AS-AP

3.1.3. PC with Chromeleon (version 7.2 or greater) software

3.1.4. Dionex AS 18-4 µm 2x150 mm column (Dionex P/N 076036). A spare column should be kept on hand.

3.1.5. Dionex IonPac AG18-4µm RFTIC 2x30 mm Guard (P/N 076037).

3.1.6. Thermo Scientific KOH Eluent Generation Cartridge (Dionex P/N 075778)

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3.1.7. Dionex AERS 500 2mm suppressor (Dionex P/N 082541)

3.1.8. Thermo Scientific 1 mL AS-AP syringe (Dionex P/N 074307)

3.1.9. Dionex CRD 200 200 µL (Dionex P/N 062986)

3.2. Consumable parts used by the instrument which are either disposable after one use or frequently replaced:

3.2.1. Thermo Scientific 1.5 mL Polyprop vials (Dionex P/N 079812 100 pk)

3.2.2. Thermo Scientific PS 10 mL vials with cap (Dionex P/N 074228 100 pk)

3.2.3. High pressure VIPER PEEK fittings (P/N 22000-98001)

3.2.4. High Pressure VIPER ferrules (P/N 043276)

3.2.5. Assortment of PEEK tubing

3.3. Glassware and pipets for preparing samples, spikes, reagents, and standards.

3.4. 10 mL disposable syringes and 25 mm syringe filters (0.45 µm pore size), for prefiltering samples

3.5. Tools for instrument maintenance (at least adjustable wrenches, hex keys, and a sharp probing tool)

3.6. Parafilm

3.7. A properly calibrated analytical balance, spatula, and weigh boats for weighing dry standards and reagents

3.8. Disposable polystyrene beakers to use in preparing dilutions or spikes

3.9. Transfer pipettes

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

Note: Before preparing reagents or standards, or running samples, please read the safety notes in the safety/hazardous waste section of this SOP and the MSDS for the chemicals used in this analysis.

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 standard and reagent sheets submitted with each analysis folder. Certificates of Analysis for each purchased standard are maintained in a binder in C307. If available, a Certificate of Analysis for each purchased reagent is also maintained in the REAGENT_PREP 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.

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4.1. Deionized (DI) water.

4.2. Eluent is automatically generated through the KOH Eluent Generator cartridge using DI water.

4.3. Other solvents (HPLC or ACS grade) for cleaning:

4.3.1. Acetone is required for rinsing instrument reservoirs monthly.

4.4. Stock Standards – Standards are prepared from ACS certified reagent grade materials, dried at 105 ºC for 24 hours. Holding time, 180 days. Store in refrigerator:

4.4.1. Stock Chloride, 10,000 mg Cl^-/L - Dissolve 21.0282 g potassium chloride in DI water and dilute to 1 L.

4.4.2. Stock Sulfate, 10,000 mg SO_4^{2-}/L - Dissolve 18.1402 g potassium sulfate in DI water and dilute to 1 L.

4.4.3. Stock Nitrate, 1000 mg N/L - Dissolve 3.6091 g potassium nitrate in DI water and dilute to 500 mL.

4.4.4. Stock Bromide, 100 mg Br^-/L - Dissolve 0.1288 g sodium bromide in DI water and dilute to 1 L, or use a purchased certified Bromide standard (Ricca Chemical, cat# 1180-4, **1000 mg Br^-/L**).

Note: If using the 1000 mg Br^-/L purchased standard from Ricca, adjust volumes used to prepare working standards accordingly. See Table 1 below.

4.4.5. 100 ppm Working Bromide Standard - Using a calibrated Eppendorf micropipet, place 1 ml of 1000 ppm purchased certified standard from Ricca in volumetric flask partially filled with DI water and dilute to mark with DI water. Holding time, 30 days. Store in the refrigerator.

4.5. Calibration Standards (Table 1) - Using a calibrated Eppendorf micropipet, place the appropriate volume of stock (4.4.) for each analyte in a 250 mL volumetric flask partially filled with DI water and dilute to mark with DI water. Standards can also be prepared through autodilution. Holding time, 30 days. Store in the refrigerator.

Table 1.

	Chloride		**Sulfate**		**Nitrate**		**Bromide**	
STD1	0.25 ppm	6.25 µL	0.25 ppm	6.25 µL	0.080 ppm	20.0 µL	0.030 ppm	75.0 µL*
STD2	0.50 ppm	12.5 µL	0.50 ppm	12.5 µL	0.10 ppm	25.0 µL	0.040 ppm	10.0 µL
STD3	1.0 ppm	25.0 µL	1.0 ppm	25.0 µL	0.20 ppm	50.0 µL	0.060 ppm	15.0 µL
STD4	2.5 ppm	62.5 µL	2.5 ppm	62.5 µL	0.50 ppm	125 µL	0.10 ppm	25.0 µL
STD5	5.0 ppm	125 µL	5.0 ppm	125 µL	1.0 ppm	250 µL	0.20 ppm	50.0 µL
STD6	12 ppm	300 µL	8.0 ppm	200 µL	1.0 ppm	250 µL	0.40 ppm	100 µL
STD7	30 ppm	750 µL	20 ppm	500 µL	2.5 ppm	625 µL	0.60 ppm	150 µL
STD8	120 ppm	3000 µL	80 ppm	2000 µL	10 ppm	2500 µL	1.0 ppm	250 µL

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Note: Bromide may be added to the standards for method development purposes only.

* Uses Bromide 100 ppm Working Standard (4.4.5.)

4.6. Combined Working Standards (Table 2) - Using a calibrated Eppendorf micropipet, place the appropriate volume of stock (4.4.) in a volumetric flask partially filled with DI water and dilute to mark with DI water. Holding time, 30 days. Store in the refrigerator.

Table 2.

	Volume Prepared	Chloride		Sulfate		Nitrate	
MDL	**250 mL**	0.20 ppm	5.0 µL	0.20 ppm	5.0 µL	0.05 ppm	12.5 µL
PQCK5	**500 mL**	0.50 ppm	25 µL	0.50 ppm	25 µL	0.10 ppm	50 µL
CHCK5	**1000 mL**	10 ppm	1000 µL	5.0 ppm	500 µL	0.80 ppm	800 µL
CHCK2	**1000 mL**	20 ppm	2000 µL	20 ppm	2000 µL	0.80 ppm	800 µL
CHCKHI4	**250 mL**	120 ppm	3000 µL	80 ppm	2000 µL	10 ppm	2500 µL

4.7. Prepare the Interference Check Standard (INT_STD) and CHCK1 in 250 mL volumetric flasks using the indicated amounts of stock standard (4.4). Holding time, 30 days for both standards. Store in the refrigerator.:

	CHCK1		INT_STD	
Chloride	2.5 ppm	62.5 µL	2.5 ppm	62.5 µL
Sulfate	2.5 ppm	62.5 µL	2.5 ppm	62.5 µL
Nitrate	1.0 ppm	250 µL	1.0 ppm	250 µL
Bromide	0.10 ppm	25 µL	0.10 ppm	25 µL
Fluoride	N/A	N/A	100 ppm	2500 µL
Nitrite	N/A	N/A	1000 ppm	250 µL
Phosphate	N/A	N/A	1000 ppm	250 µL

4.8. A certified quality control (ALTQC) sample prepared following the manufacturer's instructions. See the manufacturer's certificate for concentration and expiration date. Store in the refrigerator.

5. SAMPLE PREPARATION PROCEDURE (SEE DEP SOP NU-033)

5.1. All samples are stored on ice or at ≤ 6 °C (not frozen). Samples to be analyzed for chloride **only** do not require the above storage.

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5.2. Samples should be analyzed for chloride or sulfate within 28 days of collection date. Samples should be analyzed for nitrate within 48 hours of collection. If samples are analyzed outside these hold times, results must be reported with the Q qualifier code.

5.3. Solid matrix samples first undergo an aqueous extraction procedure according to DEP SOP NU-044.

5.4. Remove the samples from the refrigerator following the proper protocols to preserve the samples chain of custody. Allow the samples to come to room temperature before pouring them into the autosampler vials.

5.5. Samples for the IC are pre-diluted based on conductivity. See section 6.3. for detailed instructions.

5.6. For each batch –

- One batch may contain up to 20 samples.
- Prepare one matrix spike for every 10 samples. Add the appropriate amount of spiking solution (Stock Standards 4.4.) into a 10 mL portion of a selected sample. Prepare either a duplicate sample, if the components are known to be above the practical quantitation limit (PQL/PQCK5), or one matrix spike in duplicate.
- Prepare one laboratory fortified blank (LFB) following the same procedure substituting DI water for the sample.
- Effluents are batched separately. Make sure that an effluent sample is spiked to represent the batch.

6. SAMPLE ANALYSIS/QUANTITATION

6.1. Preparing the Instrument:

6.1.1. Ensure that the pump, eluent generator compartment, DC compartment, and the autosampler are powered on.

6.1.2. The DC compartments are kept at a constant temperature of 30 C. The compartment doors should be kept shut unless doing maintenance and not opened at all during analysis.

6.1.2. Turn on the computer and open Chromeleon 7.

6.1.3. Connect all the compartments to Chromeleon 7.

6.1.3. Perform the weekly maintenance detailed in section 10.2.

6.1.4. Turn the pump on to a lower flow rate.

6.1.5. Once the system pressure exceeds 200 psi and starts to stabilize, turn on the eluent generator, CR-TC, and suppressor in that order.

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6.1.6. Once temperature of the DC compartment stabilizes at 30 C, change the pump flow rate, eluent concentration, and suppressor to the first of the method parameters (Table 3). The pump flow rate should be increased in increments of 0.5 mL/min.

Table 3.

	Time (min)	Value
Temperature	All	30.0 C
Flow Rate	All	0.38 mL/min
Eluent Concentration	Initial	17.0 mM
	0 → 5.5	17.0 mM
	5.5	30.0 mM
	5.5 → 6.0	30.0 mM
	6.0	60.0 mM
	6.0 → 7.0	60.0 mM
	7.0	17.0 mM
	7.0 → 10.0	17.0 mM
Suppressor	All	57 mA

6.2. Preparing a Sample Queue:

Note: It is important that all system folders, sequences, methods, and processing methods are kept together. Do not mix system 1 with system 2 or vice versa.

6.2.1. Click the “Data” Module.

6.2.2. For the system to be used, create a sequence by right clicking that system’s folder (System_1 or System_2) and selecting the month.

6.2.3. Right click the month’s folder and click “New Sequence..”

6.2.3. In the Sequence pop up, click the corresponding system then “Next.”

6.2.4. Leave the box for “Pattern for Injection Name” blank, Insert the number of vials approximately needed, set “Injections per Vial” to 1, pick a vial starting position (Typically RA1), and the injection volume to 2.5 µL. Then click “Next.”

6.2.5. Click “Browse” for the instrument method. In the corresponding system, find the “Template” folder and insert the method (for example: S1_17 G in System 1’s folder and S2_17G in System 2’s folder).

6.2.6. Repeat 6.2.5. for the processing method (S1_Anions for System 1 and S2_Anions for System 2) and the Report template.

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6.2.7. Name the sequence with the format YYMMDD_S#.

6.2.8. Beginning with the calibration curve, enter your run sequence.

An example run sequence includes:

CAL STDs STD1-STD8 (4.5.)
BLK_carryover (DI water, 4.1.)
INT-STD (Interference check standard, 4.7.)
CHCK1 (4.7.)
MDL (4.6.)
PQCK5 (4.6.)
ALTQC (4.8.)
CHCKHI4 (4.6.)
CHCK5 (4.6.)
BLK (DI water, 4.1.)
Batched samples (20 max.), including an MBLK, an LFB**.** (For matrix spikes and spiking instructions see section 5.6.) Bracketing checks and blanks must be added for every 10 samples.
CHCK2 or CHCK5 (4.6.)
BLK (DI water, 4.1.)

6.2.9. In the "Type" column, set the calibration standards (4.5) to "Calibration Standard."

6.2.10. The "Level" column indicates whether a given vial contains a calibrant. Set each standard to its corresponding level. (STD 1 is level 1, STD 2 is level 2, etc.)

6.3. Sample Preparation:

6.3.1. Samples for the IC are pre-diluted based on conductivity or salinity. Use Table 4 as a guide. This can be done either manually or through auto-dilutions.

Table 4.

Conductivity (µS/cm)	Salinity (PPTh)	Dilution Factor
0-449	0-0.20	nil
450-999	0.21-0.40	2
1000-2999	0.41-2.0	10
3000-9999	2.1-4.3	20
10,000-14,999	4.4-8.6	40
15,000-19,999	8.7-10.7	50
20,000-29,999	10.8-21	100
30,000+	22-43	200

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Samples to be analyzed for nitrate only should not generally be diluted according to conductivity. If time before sample expiration permits, these samples should be screened at a 1:10 dilution factor to determine the most appropriate dilution factor.

6.3.2. Samples with particulates should be pre-filtered with a 0.45 µm syringe filter. If sample needs to be filtered, the MBLK should also be filtered.

6.4. Running over multiple days:

6.4.1. The IC is capable of running over multiple days, as long as it does not run out of DI water. These bottles can be easily re-filled while the run is in progress.

6.4.2. If running over multiple days, perform all of the daily maintenance indicated in 10.3 at the beginning of every day.

6.4.3. When running over multiple days, remember to run the PQL standard every 24 hours (see 8.6.). If it fails, shut down the run and begin again with a new calibration curve.

6.4.4. Whenever an injection has not been made for an hour or more, or whenever the pump stops run the QC indicated in section 8.7. before proceeding with new injections.

6.4.5. If a run requires more cups than the maximum capacity of the autosampler, simply begin again at position 1 on the autosampler – replace the injected cups with new cups.

6.5. Shutdown:

6.5.1. The IC pump, EG, suppressor, and temperature should not be shut off day to day. Instead, the parameters are lowered to conserve resources and ensure a quickly stabilizing baseline the next day.

6.5.2. If there is a long weekend or the IC does not need to be ran for 3 days, the IC can be turned off in the following order: Suppressor → CR-TC → EG → Pump.

6.6. Quantitation/Calculations:

6.8.1. The instrument will give analysis results for: Sulfate in mg SO_4^{2-}/L, Chloride in mg Cl^-/L, and Nitrate in mg N/L.

6.8.2. The QC Manager calculates recoveries for spikes and QC standards as well as relative percent differences (RPD) for duplicate samples and spikes.

6.8.3. The individual calibration standards are prepared according to section 4.4. The calibration is calculated by the instrument software for each component and is included in the analysis report. It also includes a correlation coefficient to evaluate the fit. The fit is cubic.

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7. POST-RUN PROCEDURE

7.1. After the sequence has finished, double click the first chromatogram with the analytes. This will open the "Data Processing" tab.

7.2. In "Presets," click the "Calibration and Results" window. Click the first analyte peak in the chromatogram and then click the "Calibration" tab at the bottom. Check the correlation coefficient.

7.3. Go through each chromatogram to check for peak shape, integration, and peak results. The tab "Peak Results" at the bottom will also display the results.

7.4. Click the "Summary" tab at the bottom to view all the results in the sequence for the selected analyte.

7.5. After checking all the chromatograms, click "Report Designer" under "Data Processing" to access the needed printouts. The report must contain:

- The sequence.
- A printout of all calibration curves.
- The chromatograms of all the initial QC.
- The peak results.

7.6. To print the method, click the "Instrument Method" tab above "Data Processing."

7.7. Generate an export file for QC Manager by clicking the upper left circle in "Report Designer" and selecting "Export." Select "Current sequence," pick the folder at \\FLDEP1\DEAR\LABS\lab3\Chem_Data\Inorganic\Nutrients\Dionex_IC\. Select the excel format needed for QC Manager. Give the file a name based on the date the run started, using the format YYMMDD_S#_B, where # indicates the instrument used and, if necessary, poscripts B, C, etc indicates the second, third, etc. run of the day. Append each file with "_CS" for chloride and sulfate and "_N" for nitrate.

7.8. Export the chromatogram files to the Nutrients network folder. In the "Data" module, click the sequence then File → Send to. Select the appropriate folder for the IC instrument used at \\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\Dionex_IC\Data\S1 and \Nutrients\ Dionex_IC\Data\S2\. Place the directory into the appropriate subdirectory for month and year. Uncheck the "Include 3D and MS raw data" option and then click "Start."

7.9. The amount of manual reprocessing will be minimal. Try to manually reprocess as few chromatograms as possible. If a chromatogram must be manually reprocessed, note this on the sequence report, along with an explanation of the reason. Print out a copy of the chromatogram with and without manual reprocessing applied and include them in the report. For the chemistry section's policy on manual integration, see DEP SOP CM-018.

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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, 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. A new calibration is performed at the beginning of every analytical run. The working calibration range is verified every time a new calibration is performed. The correlation coefficient for every component reported must be equal to or larger than 0.995. The acceptance limits for the recoveries for each calibrant above the PQL are 90 - 110 %.

8.3. Certain QC samples must be run at the beginning of every run sequence. These are:

8.3.1. A second source (ALTQC, 4.8.). The acceptance limits for the recovery of the ALTQC are 90 - 110 %.

8.3.2. A PQL-level check standard (4.6.). Acceptance limits for the recovery of the PQL are 70 - 130 %.

8.3.3. A MDL-level check standard (4.6.). There are no defined acceptance limits for MDL-level standards, but all analytes must be detected and each must be readily distinguishable from the PQL-level standard for that analyte.

8.3.4. An Interference Check Standard (4.7), which has acceptance limits of 85 - 115 % recovery. This should be run with a CHCK1 (4.7), which has the same levels of each analyte. That way, if there is a failure in the Interference Check Standard, the analyst can tell if it is related to interference or a calibration problem. The acceptance limits on the CHCK1 are 90 - 110 % recovery.

8.3.5. A blank to check for carryover. This is simply DI water, and is run immediately after the highest calibration standard. The carryover blank has the same acceptance limits as other blanks.

8.3.6. A CCV with the same values as the upper limit of calibration, the CHCKHI4 (4.6.). This has the same acceptance limits as other CCVs, 90 - 110 % recovery.

8.4. Each run should include a bracketing CCV and a CCB (DI water) every 10 samples (i.e. every 12^{th} cup).

8.4.1. For the CCV, alternate the CHCK2 and CHCK5 standards (see 4.6.). Acceptance limits for the recovery of the check standards are 90 -110 %.

8.4.2. Results for all blanks (CCB/BLK) should be less than the MDL and greater than –MDL.

8.5. Certain QC must be run for every batch of up to 20 samples:

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8.5.1. A method blank (MBLK). This consists of DI water that is prefiltered with a syringe filter if samples are filtered.

8.5.2. Precision must also be demonstrated so run either a duplicate sample or duplicate matrix spike with each batch of 20 samples to demonstrate that the relative percent difference (RPD) is < 20 %.

8.5.3. For each batch of 20 samples, two matrix spikes of different samples and an LFB must also be analyzed to ensure that analyte recovery is within acceptable ranges (see section 5.6). Follow the standard spiking protocol for duplicates and spikes. Acceptance limits for matrix spike recoveries and LFB recoveries are 90 - 110 % for aqueous matrices, and 80 - 120 % for solid matrices.

Spike a 10 mL aliquot of sample (or DI water for LFBs) with all analytes for each level.

Spike N3:

Analyte	Spike Level (mg/L)	Stock Conc. (mg/L)	Amount of Stock (µL)
Sulfate/Chloride	30	10,000	30
Nitrate	5.0	1000	50

8.6. The IC can run over more than one day if the calibration range is checked and confirmed to be acceptable. This can be done by running a PQL standard at least every 24 hours.

8.7. If the instrument has not injected a sample for an hour or more, or the pump has shut down and been re-started, the calibration must be checked before running new samples. This is done by running a CCV, a PQL standard, and a CCB.

9. DATA ARCHIVAL

9.1. 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 (IC-SO4, Cl, etc.), filename/s (YYMMDD_S#-LL, where # indicates the instrument used and -LL indicates the components reported in the run. CS stands for sulfate and chloride, and N for nitrate), and initials of the analyst.

9.2. Each report folder should contain the following items - the calibration reports with method parameters, results report(s), bench sheet(s), reagents and standard sheets, chromatograms for initial QC and a copy of the data and QC assessments uploaded from QC Manager to LIMS at the time of test authorization.

9.3. 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.

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9.4. IC instrument data excel transfer files for upload to QC manager are stored in electronic format located on the Nutrients drive: \\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\Dionex_IC\.

10. MAINTENANCE

Note: All maintenance must be recorded in the instrument maintenance manual.

10.1. Keep the instrument clean and free of dust as much as possible. If eluent or sample is spilled, be sure to clean it up right away.

10.2. Weekly maintenance. Perform the following maintenance procedures before you calibrate the instrument for the first time in a calendar week:

10.2.1. Make sure that eluent generation cartridge doesn't need to be changed.

10.2.3. Empty and rinse the DI water reservoirs that go to the pumps and AS-AP. Refill.

10.2.4. Prime the pump:

Open the corresponding system's purge valve, go to the "Pump" tab for that system, click "Prime," then "Execute despite warning." Wait a few minutes until all the bubbles go to waste. Click "Stop," **close the purge valve**, and turn the pump on to 0.15-0.20 mL/min.

10.2.5. Prime the AS-AP:

Ensure there are no bubbles in the syringe. Click the "Sampler" tab. Then "Wash Sample Loop" with 1000 µL. Then "Prime Syringe" for 10 cycles. Then "Wash Needle and Fluidics" for 1000 µL. Check there are no bubbles in the syringe. If so, manually prime the syringe.

10.2.6. Click "Monitor Baseline" and wait for a stable baseline.

10.3. Daily maintenance for ongoing runs. Perform the following maintenance for every day in a calendar week after the first day that the instrument continues to run:

10.3.1 Top off DI water reservoirs.

10.3.2. Check for leaks or unusually low pressure.

10.3.3. Check for air bubbles in syringe.

10.3.4. Click "Monitor Baseline" and watch for a stable baseline.

10.4. Routine replacement of parts:

10.4.1. The Eluent generator cartridge and corresponding pieces should be replaced when EGC % remaining is low.

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10.4.2. The analytical column should be replaced when the pressure gets close to the maximum (5000 psi) or the peak resolution and baseline is too poor to continue use.

10.5. If any significant changes are made to the system, such as a new column, check the analytes' retention time and any interfering anion retention times by running the interference check standard. All peaks should be clearly resolved and well within the established retention time windows.

10.6. Record in the maintenance log any maintenance carried out. Write down the serial numbers of all parts removed from or installed on the system. Also, place all service visit receipts in the maintenance log.

11. SAFETY/HAZARDOUS WASTE MANAGEMENT/POLLUTION PREVENTION

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

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

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

11.4. The IC analysis does not normally generate hazardous waste, so the eluent and sample waste can go down the drain. The Dionex may be set up to flush waste directly into a sink, or the waste can be directed into a plastic carboy that is emptied as needed.

12. REFERENCES/DEFINITIONS/VALIDATION DATA

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. The limit of detection (LOD) is verified on every run based on the PQCK5.

12.1. Dionex AS-AP Autosampler Operator's Manual, Thermo Fisher Scientific Inc, 2012. Document No. 065361.

12.2. Dionex IonPac AS18-4µm Column Product Manual, Thermo Fisher Scientific Inc, 2014. Document No. 065499-02.

12.3. Dionex ERS 500 Suppressor, Thermo Fisher Scientific Inc, 2017. Document No. 031956-11.

12.4. Chromeleon Manuals:

12.4.1. Chromeleon 7.2 Operator Training: Level 1, Thermo Fisher Scientific Inc, 2015. Document CM 7.2 Level 1.

12.4.2. Chromeleon 7.2 Operator Training: Level 2, Thermo Fisher Scientific Inc, 2015. Document CM 7.2 Level 2.

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12.6. EPA Method 300.0 Rev. 2.1 (1993).

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

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

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

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

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

12.12. DEP SOP NU-044, Aqueous Extraction of Orthophosphate, Nitrate, Chloride, and Sulfate from Soils and Sediments.

12.13. DEP SOP CM-018, Laboratory Policy Regarding Manual Chromatographic Peak Integration.

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

12.15. Quality Manual, Bureau of Laboratories, FDEP.

12.16. Laboratory Health and Safety Plan.

Appendix of Significant Changes

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