

**Illinois State Water Survey
Health and Environmental Applications Laboratory**

**Standard Operating Procedure
For the Determination of
F⁻, Cl⁻, Br⁻, NO₃⁻, and SO₄²⁻
by Ion
Chromatography**

(based on USEPA method 300.1 r1.0)

SOP Number AN.HEAL.IN.IC-anions

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Illinois State Water Survey
2204 Griffith Drive
Champaign, IL 61820-7463

NOTE THE HEALTH AND SAFETY WARNINGS IN SECTION 4.0

Prepared by: Margarita Bargon Date: 12/19/2024
Rita Bargon, QA Officer

Approved by: Evan Rea Date: 12/19/2024
Evan Rea, HEAL Director

The following individuals (in addition to those listed on the cover) have read/reviewed this SOP:

Tatyana Grandt *Tatyana Grandt* 01/03/2025
 Name (printed) Signature Date

George Barton *George Barton* 01/07/2025
 Name (printed) Signature Date

 Name (printed) Signature Date

 Name (printed) Signature Date

 Name (printed) Signature Date

 Name (printed) Signature Date

 Name (printed) Signature Date

Beginning Revision	Ending Revision	Revision Date	Changes
12.0	12.1	4/26/22	Corrected calibration curve procedure in 14.1.2, added 17.9 concerning oxalate and bromide, and corrected data export location
12.1	13.0	9/15/23	Major changes to formatting and section order. QC section 14 overhauled to be more consistent with other analyses.
13.0	13.1	7/22/24	Updated QC scheme, added reagents and instructions for running surface water samples for NEON Water Chemistry project
13.1	13.2	10/8/24	Added Fluoride to title and text where it was left out.

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1.0 Scope & Applicability

Fluoride (F⁻), Chloride (Cl⁻), sulfate (SO₄²⁻), bromide (Br⁻), and nitrate (NO₃⁻) are determined by Thermo Scientific Autosuppressed Ion Chromatography (IC) for the wet deposition samples. The Method Detection Limit for NO₃⁻ is 0.006 mg/L, for SO₄²⁻ is 0.006 mg/L, for Cl⁻ is 0.004 mg/L, for F⁻ is 0.04 mg/L, and for Br⁻ is 0.002 mg/L.

2.0 Summary of Method

This ion chromatography (IC) method closely follows EPA Method 300.1. This ion chromatography method combines conductivity detection with the separation capabilities of Thermo Scientific AG18/AS18 columns. A 25 µL aliquot of sample is pumped through an ion exchange column. Because different ions have different migration rates, the sample ions of interest (F⁻, Cl⁻, Br⁻, SO₄²⁻, NO₃⁻) elute from the column as discrete bands. Each ion is identified by its retention time. The sample ions are selectively eluted off the analytical column and into an anion self-regenerating suppressor (ASRS). The eluent ions are neutralized, and the sample ions are converted to their corresponding strong acids which are detected in a conductivity cell. The ion chromatographs are calibrated with standard solutions containing known concentrations of anions. The raw peak area data is collected by the computer. The electronically produced Chromatogram is a picture of the raw data. The Chromeleon software is used to control each IC system and to calculate the concentration of the anions for each sample using the raw data. IC operating conditions are as follows:

Guard column - Thermo Scientific AG18 (for ICS-5000) and AG18-4 µm (for Integriion)

Separator column - Thermo Scientific AS18 (for ICS-5000) and AS18-4 µm (for Integriion)

Anion Suppressor - Thermo Scientific ASRS - Ultra II

Detector - Thermo Scientific Conductivity Detector CD1 cell (for ICS-5000) and Integriion CD Conductivity Detector (for Integriion)

Eluent - 31 mM Potassium Hydroxide

ASRS - Ultra II current = 87 mA

Column Temp = 30°C

Cell Temp. = 35°C

Sample loop - 25 µL

Flow rate - 1.0 mL/min

3.0 Definitions

ACS American Chemical Society

CCB Continuing Calibration Blank. HEAL uses deionized water as a blank, named FB.

CCV Continuing Calibration Verification. HEAL uses several different CCVs for wet deposition analysis:

FH – a solution targeting the high end of the calibration curve

FM- a solution targeting the middle of the calibration curve

FL- a solution targeting the low end of the calibration curve

DI	Deionized (water) at 18.0 Mohms-cm or higher
ICV	Initial Calibration Verification. HEAL uses a simulated rain sample as an ICV: FR50- a solution with target analyte concentrations at the 50th percentile of the NTN network results.
MDL	Method Detection Limit
HDPE	High Density Polyethylene
LIMS	Laboratory Information Management System
QA	Quality Assurance
QC	Quality Control
R²	Coefficient of Determination reflecting the deviation of the measured data points from the calibration curve
SDS	Safety Data Sheet
SOP	Standard Operating Procedure

4.0 Health & Safety Warnings

- 4.1 Always wear eye protection in the laboratory.
- 4.2 Food, drinks, and smoking are not allowed in the laboratory.
- 4.3 Safety Data Sheets (SDS) applicable to this SOP can be found online by using the University of Illinois Division of Research Safety (DRS) website: <https://www.drs.illinois.edu/Programs/SafetyDataSheets>.
- 4.4 The Illinois State Water Survey Chemical Hygiene Plan covers the ISWS laboratory safety program, including, but not limited to, personal protective equipment used, control equipment inventory and operations (such as vented hoods), employee training programs, medical programs, and safety. The ISWS Chemical Hygiene Plan is available at <https://go.illinois.edu/ISWS-Chemical-Hygiene-Plan>.
- 4.5 The University of Illinois DRS has a laboratory safety guide available at <https://www.drs.illinois.edu/site-documents/LaboratorySafetyGuide.pdf>. The ISWS has their own laboratory safety manual, available at <https://go.illinois.edu/ISWS-Laboratory-Safety-Manual>.
- 4.6 The HEAL practices pollution prevention, which encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. The quantity of chemicals purchased should be based on the expected usage during its shelf life and disposal cost of unused material.
- 4.7 Laboratory waste management practices must be consistent with all applicable rules and regulations. Excess reagents and samples and method process wastes should be characterized and disposed of by DRS. It is the responsibility of the user of this method to comply with relevant disposal and waste regulations.
- 4.8 The HEAL has listed known health and safety warnings for this SOP, but this list should not be assumed to comprise all health and safety issues.

5.0 Cautions

- 5.1 Wear eye protection and gloves when handling the Eluent Generator Cartridge (EGC).
- 5.2 Do not cap EGC waste container; small amounts of oxygen and hydrogen gas are produced.
- 5.3 Wait 15 seconds after turning off equipment before turning it on again. This will prevent damage to pump circuitry and components.
- 5.4 Electrical precautions: turn off power and unplug electrical cords when replacing fuses, circuit board, and other electrical parts.

6.0 Interferences

- 6.1 To prevent interferences use chemicals of high purity 99% or better.
- 6.2 Air bubbles in the eluent can cause the pump to lose prime as well as noise in the detector cell.
- 6.3 Use DI with a resistivity of 18.0 megohms-cm or greater and a 0.45 µm point of use filter to help prevent contamination problems.
- 6.4 Occasionally a peak with a retention time similar to Cl⁻, NO₃⁻, Br⁻, or SO₄²⁻ interferes with peak integration. The sample can be diluted to separate the peaks better, or a sample aliquot can be spiked to determine the correct peak. If a Cl⁻, NO₃⁻, Br⁻, or SO₄²⁻ peak cannot be completely resolved, notify the Laboratory Director before the data is released.

7.0 Personnel Qualifications

Analysts in training must complete at least five days of training with an experienced analyst or a manufacturer's training course, and a satisfactory demonstration of capability before analyzing routine samples.

8.0 Equipment and Supplies

- 8.1 Equipment
 - 8.1.2 Integriion Ion Chromatograph, purchased
 - 8.1.3 Thermo Scientific ASDV autosampler
- 8.2 Supplies (purchased from Fisher Scientific)
 - 8.2.1 AG18/AS18 (AG18-4µm/AS18-4µm) Guard/Separator Columns
 - 8.2.1.2 ASRS (ULTRA II - 4mm) Anion Suppressor
 - 8.2.1.3 Sample vials with filter caps from Thermo Fisher Scientific. Vials part # 038008, filter cap part # 038009.
 - 8.2.1.4 Piston seals

- 8.2.1.5 Peak Tubing
- 8.2.1.6 Eluent Generator Cartridge - Anions (EGC)
- 8.2.1.7 Continuously Regenerated Anion Trap Column (CR-ATC)

8.3 Other Supplies

8.3.1 Adjustable pipettes and tips

8.3.2 Class A Volumetric flasks

8.4 Chemicals and Solutions

Certified stock solutions (1000 mg/L) are purchased from AccuStandards for calibration standards and SPEXCertiPrep for QCs.

8.4.1 QC Preparation

Prepare Calibration Standards using DI water with a specific resistance of 18.0 megohms-cm, use only class A volumetric flasks. See the Table 1 below for specific amount of stock used for each standard for the Wet Deposition samples analyses. Record the lot number, expiration date, and manufacturer of each stock solution in the IC logbook when preparing new QCs. The QC concentrations and necessary volumes added to achieve the concentrations desired for the Water Chemistry Project (surface water samples) are shown in Table 2.

Table 1 – Standards Concentrations and Stock Volumes for Standards Preparation for Wet Deposition sample analysis

Standard	Volumetric Flask Size	Cl ⁻ Value mg/L	Amount of Cl ⁻ Stock to use	NO ₃ ⁻ Value mg/L	Amount of NO ₃ ⁻ Stock to use	SO ₄ ²⁻ Value mg/L	Amount of SO ₄ ²⁻ Stock to use	Br ⁻ Value mg/L	Amount of Br ⁻ Stock to use
S0	1 L	0.015	15 µL	0.015	15 µL	0.015	15 µL	0.015	15 µL
S1	1 L	0.025	25 µL	0.050	50 µL	0.050	50 µL	0.025	25 µL
S2	1 L	0.050	50 µL	0.100	100 µL	0.100	100 µL	0.050	50 µL
S3	1 L	0.100	100 µL	0.200	200 µL	0.200	200 µL	0.100	100 µL
S4	1 L	0.200	200 µL	0.500	500 µL	0.750	750 µL	0.200	200 µL
S5	1 L	0.500	500 µL	1.00	1.00 mL	1.500	1.5 mL	0.500	500 µL
S6	500 mL	1.000	500 µL	2.50	1.25 mL	2.500	1.25 mL		
S7	500 mL	1.500	750 µL	4.00	2.00 mL	4.000	2.00 mL		
S8	500 mL	2.500	1.25 mL	5.00	2.50 mL	5.000	2.50 mL		
S9	500 mL	4.000	2.00 mL	6.00	3.00 mL	6.000	3.00 mL		

8.4.2 Standards 0-6 are used to calculate samples that fall in the range 0 - 1.000 mg/L for Cl⁻, 0 – 0.500 mg/L for Br⁻ and 0 - 2.500 mg/L for NO₃⁻ and SO₄²⁻. Standards 6-9 are used to calculate samples that fall in the range 1.000-4.000 mg/L for Cl⁻ and 2.500 - 6.000 mg/L for NO₃⁻ and SO₄²⁻.

8.4.3 A standard spiked with oxalate is analyzed every run after the calibration to define oxalate and bromide curve. In a 60 mL bottle, add 50 mL of standard 5 and spike with 1mL of oxalate stock and label as “Std 5+ox”.

8.4.4 Amount of stock solution= $\frac{\text{desired end volume (mL)} \times \text{desired concentration (mg/L)}}{\text{stock solution concentration (mg/L)}}$
 to use (mL)

Example: $1.5 \text{ mL} = \frac{500 \text{ mL} \times 3 \text{ mg/L}}{1000 \text{ mg/L}}$

Table 2 - Calibration Standards and Stock Volumes for the Water Chemistry samples.

Standard	Volumetric Flask Size	Cl ⁻ Value mg/L	Amount of Cl ⁻ Stock to use	F ⁻ Value mg/L	Amount of F ⁻ Stock to use	SO ₄ ²⁻ Value mg/L	Amount of SO ₄ ²⁻ Stock to use	Br ⁻ Value mg/L	Amount of Br ⁻ Stock to use
S0	1 L	0.015	15 µL	0.015	15 µL	0.015	15 µL	0.015	15 µL
S1	1 L	0.025	25 µL	0.200	200 µL	0.050	50 µL	0.025	25 µL
S2	500 mL	0.050	25 µL	0.300	150 µL	0.100	50 µL	0.050	25 µL
S3	500 mL	0.100	50 µL	0.500	250 µL	0.200	100 µL	0.100	50 µL
S4	500 mL	0.200	100 µL	0.800	400 µL	0.750	375 µL	0.200	100 µL
S5	500 mL	0.500	250 µL	1.500	750 µL	1.500	750 µL	0.500	250 µL
S6	500 mL	1.000	500 µL	3.000	1.5 mL	2.500	1.25 mL		
S7	500 mL	6.000	3.00 mL			6.000	3.00 mL		
S8	500 mL	15.00	7.50 mL			15.00	7.50 mL		
S9	500 mL	25.00	12.5 mL			25.00	12.5 mL		

Table 3 – QC Concentrations and Stock Volumes for wet deposition samples

Standard	Volumetric Flask Size	Cl ⁻ Value mg/L	Amount of Cl ⁻ Stock to use	NO ₃ ⁻ Value mg/L	Amount of NO ₃ ⁻ Stock to use	SO ₄ ²⁻ Value mg/L	Amount of SO ₄ ²⁻ Stock to use	Br ⁻ Value mg/L	Amount of Br ⁻ Stock to use
FL	1 L	0.025	25 µL	0.50	500 µL	0.50	500 µL	0.025	25 µL
FH	500 mL	3.00	1.5 mL	5.00	2.50 mL	5.00	2.50 mL		

Table 4 – QC Concentrations and Stock Volumes for surface water samples

Standard	Volumetric Flask Size	Cl ⁻ Value mg/L	Amount of Cl ⁻ Stock to use	F ⁻ Value mg/L	Amount F ⁻ Stock to use	SO ₄ ²⁻ Value mg/L	Amount of SO ₄ ²⁻ Stock to use	Br ⁻ Value mg/L	Amount of Br ⁻ Stock to use
FL	500 mL	1.50	750 µL	1.50	750 µL	1.50	750 µL	0.50	250 µL
FM	500 mL	1.00	500 µL	0.50	250 µL	2.50	1.25 mL	0.100	50 µL

8.4.5 Calibration and quality control check solutions are stable for up to three months.

9.0 Instrument Setup and Calibration

9.1 Vials and Caps

Soak vials and caps separately in DI for at least 1 hour, and rinse 3 times. Dry in clean area in racks. Cover top of vials with parafilm while soaking and drying. Avoid contact with vials and caps to prevent contamination.

9.2 IC Start-up Procedures

Fill eluent reservoir container(s) with DI water.

Connect the pump with the system. Prime the pump - loosen the knob on the left pump head and click the **Prime** button on the pump control section of the control panel. The system will tell the operator to loosen the prime valve - click **OK**. After about 1 minute of priming, click the **Off** button for the pump, close the priming valve and click the **Pump on**, **Suppressor on** (87 mA), **Heater on** (30 °C), **Eluent Generator on** (31 mM), **CR-TC on**, and **Cell Temp on** (35 °C) buttons. Flow rate should be 1.0 mL/min.

9.3 Check the IC for any leaks, high pressure readings, and correct conductivity reading (< 2uS). Check that the cell temperature is set to 35°C, column temperature to 30°C, eluent concentration is set to 31 mM, and that the ASRS is on and set to 87 mA. Monitor the baseline by clicking the **Monitor Baseline** command at the top of the Instrument Chromeleon Console. Once the baseline is flat, the instrument is ready to analyze samples.

9.4 While the system is warming up, begin pouring samples. At least 2 mL of sample is required for testing. Pour calibration standards from lowest to highest concentrations, and then pour appropriate QCs (see section 14.2 for details). After the samples are poured, the sample bottles are marked with a green permanent marker.

9.5 Right before beginning analysis, record the date, background conductivity, back pressure and EGC life remaining in the ICS 5000/Integrion hardware logbook.

9.6 To start the analysis, make sure the carousel on the AS-DV is locked. If the carousel is not locked, press the **Release Carousel** button on the inside of the autosampler. Click the **Start** button from the top of the sequence.

9.7 Calibration

The calibration curve is split into high and low curves for the wet deposition samples analyses. The low curve incorporates standards 0 - 6 and has a range of 0.000 - 1.000 mg/L for Cl⁻, 0.000 – 0.500 mg/L for Br⁻ and 0.000 - 2.500 mg/L for NO₃⁻ and SO₄²⁻. The high curve incorporates standards 6 - 9 and has a range of 1.000-4.000 mg/L for Cl⁻ and 2.500-6.000 mg/L for NO₃⁻ and SO₄²⁻.

9.7.1 After the calibration standards are analyzed, check the coefficient of determination values for the calibration curves. To check the calibration curves:

- In Chromeleon Console double click on the run to work with,
- Double-click on **Standard 5** of the set of standards.
- Click on **Processing** tab in the ribbon, then click on **Detection Parameters**.
- Adjust the inhibit off/on parameters. The inhibit off line goes in front of the peak and the inhibit on line goes after the peak.
- Click on Data Processing Home tab in the ribbon, then choose **Calib & P.M.** tool in the ribbon → **Component Table**.
- In the **Component Table** insert the retention times for all ions.
- In the **Calibration** tab ensure that the standards of interest are checked (0-6 for low curve, 6-9 for high curve). Also, ensure that all concentrations for the standards are correct in the table. Click **Save** button at top of page.
- Click on **Results** in the ribbon.
- Go to **Calibration** tab at bottom of page.
- Check the linearity of the low and high curve for all ions. Ensure that all analytes have a $r^2 > 0.9995$.

10.0 Sample Collection, Handling and Preservation

10.1 The IC operator should be careful when handling vials, caps, samples and standards to prevent standard and sample contamination.

10.2 Always use the cover on the samplers to keep particulate matter out of the samples.

10.3 Samples are refrigerated at 4°C. Samples are always refrigerated, except to pour them. Time and frequency of sample pouring should be minimized.

11.0 Sample Preparation & Analysis

11.1 Retrieving Lab numbers for Daily Runs and Preparing Sequences

11.4.1 Create run sequences in Chromeleon by first opening the template sequences (found in the Chromeleon Console (see Appendix B) under the data option, Chromeleon Local Directory, in the folder for the current month. Scan sample bar codes into the template to create the run sequence.

11.4.2 Add appropriate QCs at the beginning, every 10 samples and at the end of the run. See details in section 14.2

11.4.3 When edits to the temp file are complete, click File, Save As and save the sequence in the folder for the current month. Use the format MMDDYY as the file name.

11.4.4 Check that the appropriate program and method is being used. Integrion method is used to run wet deposition samples.

12.0 Troubleshooting and Maintenance

12.1 IC System Maintenance & Troubleshooting

- 12.1.1 Change any dirty or crimped lines. This can cause increased system pressure and poor chromatograms.
- 12.1.3 Change the pistons seals in the pump yearly, or sooner if needed, to prevent leaks & pump damage.
- 12.1.4 Change AS18/AS18-4 µm separator columns once a year, or sooner if needed. This helps to ensure good peak shapes and good peak separation.
- 12.1.5 For ASRS Ultra II cleaning and troubleshooting refer to Thermo Scientific ASRS instruction and Troubleshooting Guide. These are located in Thermo Scientific troubleshooting guide notebook. Change ASRS as needed.
- 12.1.6 Clean stator in Rheodyne valve (injection valve) when the sampler starts becoming noisier than normal (caused by blockage in stator) (Refer to Rheodyne injector operating instructions in the Thermo Scientific users guide notebook). Note: If the high-pressure alarm goes off after reassembling valve, the stator and rotor seal may be out of alignment. Make one injection to correct this.
- 12.1.7 If eluent does not measure < 1µS, check the eluent generator (EGC) % availability and suppressor age ASRS. Change EGC if less than 5% left.
- 12.1.8 If the Coefficient of Determination values start dropping below 99.95%, repeat the instrument calibration.
- 12.1.9 For electrical and other problems contact a Technical Representative of the Thermo Scientific Corporation at (800) 346-6390. The technical representative arranges service calls.
- 12.2 Integrion Methods Maintenance manual <https://assets.thermofisher.com/TFS-Assets/CMD/manuals/Man-22153-97003-IC-Integrion-Man2215397003-EN.pdf> (see 18.9).

12.2.1 Change retention times in IC methods when columns are changed.

12.2.2 Adjust data events and end run time as needed, i.e. when column ages, retention times shorten and run time can be shortened correspondingly.

12.3 Autosampler Maintenance

Change sampling tip and tubing yearly (refer to Thermo Scientific autosampler manual).

13.0 Data and Records Management

Data acquisition parameters are located in Chromeleon Studio (see Appendix D). The software, along with the selected Data Processing file, processes and plots incoming data. The Data Processing file calculates the analytical concentrations

and saves the chromatogram and report. The data is reported to three decimal places. The raw data and report are automatically saved on the computer.

13.1 Calibration curve

The calibration curve type is “quadratic, Add Zero, 1/A” and the evaluation type is “area”. Select 1/amount (x) for proportionate weighing of standards and include point (0.0) for curve fitting. These parameters are used to construct a calibration curve for each ion.

13.2 Sample Chromatogram Review

- **Double click** on the first sample in a daily sequence to view chromatogram. Go to **Peak Results** tab at the bottom of page.
- Check the chromatogram for proper peak shapes and any mismarked peaks. All peaks should be properly integrated. Special attention should be taken when looking at the bromide peaks.
- Use the arrow buttons to view the next chromatogram.
- While checking all chromatograms, also check QC values. Compare QC values with control limit data.
- Check for samples that need dilutions: any samples with values greater than the highest standard need to be diluted.

13.3 Creating Excel files (.xls) to export data to LIMS

If data passes all quality control specifications, make two Excel files – one for low curve A and the other – for high curve B. While in an open chromatogram window, select **Report Designer**. Use month \day\year\A or B sys# for file name (example, “021218ASys5.xls”), where “A” is the low curve (standards 0-6) and “B” is the high curve (standards 6-9). Click the Chromeleon icon at the top left of the screen and select **Export**. The parent folder is [\\pri-fs1\HEAL\Data\Lab Data In\IC\2022 IC Data\current month](#), and the subfolder is date (MMDDYY), A or B, SYS3, SYS4, or SYS5.

13.4 Sending data to LIMS

Data is checked in Instrumental Chemistry tool to make sure data is complete and correct before sending it to LIMS (Laboratory Information Management System).

- Double click on the **Instrumental Chemistry** icon or [\\pri-fs1\HEAL\HEAL-IT\Program Install Files\Lims\Instrumental Chemistry](#).
- Click on **Data, Load Review, IC, IC Review, Data IO, Load Review Table** from Chromeleon.
- Select the directory where the appropriate .xls file is stored. Double click on the .xls file to import.
- Click on the NUMBER column to sort the data. Review the QC data by right-clicking each set of QC data for each analyte.
- Once all the QC data has been validated, each analyte needs to be checked for dilutions. Click on the appropriate analyte column (Cl⁻, Br⁻, SO₄²⁻, NO₃⁻) to sort the concentrations from highest to lowest. Delete any values that fall outside of the calibration curve (higher than standard 9). These samples will need to be diluted.
- When all QCs have been validated and dilutions have been held back, the data can be transferred to LIMS. Click on **Data IO, Transfer Results to LIMS Results Table**.

- To check completeness of LIMS data, click on **LIMS**, then **Query** (this brings up Query table). Enter sample range and select **Retrieve**. This will bring up data for all ions.

13.5 Daily logbooks are kept of instrument analysis, troubleshooting, repair, and maintenance. Instrument background conductivity, background pressure, and eluent generator cartridge life remaining are also recorded. This log is shared by all IC analysts. Logbooks are also used to document information about standards and QC preparations, software issues, solutions, and upgrades.

14.0 Quality Control and Quality Assurance

14.1 A calibration curve is always generated at the start of each run followed by FR50. A Coefficient of Determination of ≥ 0.9995 is required to continue with analysis.

14.2 Each run must start with the following QC samples: FR50, FB, FL, FH and MDL solutions for wet deposition sample analyses. Use FB, FL, FM and MDL solutions for the Surface water samples analyses.

14.2.1 If any of these initial QCs are outside of the acceptance limits (see table below), then they must be immediately reanalyzed before samples can be analyzed. If the reanalyzed values are still unacceptable, then the instrument should be recalibrated. The QC samples should be remade if issues persist.

14.3 After every 10 samples and at the end of each analysis, the following QCs must be analyzed: FB, FM, and a duplicate for the Water Chemistry samples. FB, FL, FH and a duplicate for the Wet Deposition samples.

14.3.1 If any of these ongoing QCs are outside of the acceptance limits, then they must be reanalyzed as soon as possible. If any samples are not bracketed by acceptable QCs, then those samples must be reanalyzed.

Table 5 - QC criteria summary table for Wet Deposition Samples.

QC	Type	Frequency	Analyte	Criteria
FR50	ICV	Every Batch	Cl	±10%
			NO3	±10%
			SO4	±10%
			Br	±20%
FL	CCV	Every Batch	Cl	±20%
			NO3	
			SO4	
			Br	
FH	CCV	Every 10 Samples	Cl	±10%
			NO3	
			SO4	
			Br	
FB	CCB	Every 10 Samples	Cl	<MDL
			NO3	
			SO4	
			Br	
Duplicate	Duplicate	Every 10 Samples	Cl	±10% if > 10 x MDL
			NO3	
			SO4	
			Br	

Table 6 - QC criteria summary table for surface water samples

QC	Type	Frequency	Analyte	Criteria
FL	ICV	Every Batch	Cl	±20%
			NO3	
			SO4	
			Br	
			F	
FM	CCV	Every 10 Samples	Cl	±10%
			NO3	
			SO4	
			Br	
			F	
FB	CCB	Every 10 Samples	Cl	<MDL
			NO3	
			SO4	
			Br	
			F	
Duplicate	Duplicate	Every 10 Samples	Cl	±10% if > 10 x MDL
			NO3	
			SO4	
			Br	
			F	

15.0 References

- 15.1 EPA Method 300.1. DETERMINATION OF INORGANIC ANIONS IN DRINKING WATER BY ION CHROMATOGRAPHY, Revision 1.0, Cincinnati, OH, U.S.EPA. 1997..
- 15.2 Integrion manual <https://assets.thermofisher.com/TFS-Assets/CMD/manuals/Man-22153-97003-IC-Integrion-Man2215397003-EN.pdf>

Appendix A Ion Chromatography Ordering Guide

NOTE: Call supplier before ordering to verify order number and current price

<u>Supplier Information</u>	<u>Part Number</u>	<u>Fisher Scientific Number</u>	<u>Name</u>	<u>Description</u>	<u>Instrument</u>
Thermo Electron North America 1228 Titan Way Sunnyvale, CA 94988 800.346.6390	039532		Autosampler Vials	PolyVials and Plain Caps 5.0 mL 250 per box	ICS 5000/Integrion
	060551		Guard Column	IonPac AG18 4mm Guard Column 4x50mm	ICS 5000
	060549		Analytical Column	IonPac AS18 4mm Analytical Column 4x250mm	ICS 5000
	076035		Guard Column	IonPac AG18 4um Guard Column 4x30mm	Integrion
	076034		Analytical Column	IonPac AS18 4um Analytical Column 4x150mm	Integrion
	082540		Suppressor	ASRS 500	ICS 5000/Integrion
	074532	NC9041253	EGC-II KOH	Potassium Hydroxide Eluent Generator Cartridge	ICS 5000
	075778		EGC-II KOH	Potassium Hydroxide Eluent Generator Cartridge	Integrion
	060477		CR-ATC	Continuously Regenerating Anion Trap Column	ICS 5000
	088662		CR-ATC	Continuously Regenerating Anion Trap Column	Integrion
	074373		Peek Tubing Ferrules	Ferrules, Double Cone	ICS 5000/Integrion
	045987		End line Filters	End line filters	ICS 5000
	036521		35u Filters	35micron filters for in-line autosampler filter	ICS 5000/Integrion
	036522		5u Filters	5 micron filters for in-line autosampler filter	ICS 5000/Integrion
	044105		InLine Filters	Hi-Pressure filter housing (10/32) with filters	ICS 5000/Integrion
			Piston	Pump Head Piston	
	AAA-061795	NC0386590	PM Kit	ICS-3000/5000 Single Pump Maintenance Kit	ICS 5000
	AAA-061796		PM Kit	ICS-3000/5000 Detector Maintenance Kit	ICS 5000
	071575	NC0334443	PM Kit	AS-DV Maintenance Kit	ICS 5000/Integrion
	064946		each	Main Piston Seal (ICS5000)	ICS 5000
	063382		each	Pison Seal Wash Seal, Priming Valve Knob Seal (ICS5000)	ICS 5000
	061830		each	Conductivity Cell for ICS 5000	ICS 5000
	063292		each	4L Plastic Bottle	ICS 5000/Integrion
	042949		each	10uL Sample Loop	ICS 5000/Integrion

Appendix A Ion Chromatography Ordering Guide (continued)

<u>Supplier Information</u>	<u>Part Number</u>	<u>Fisher Scientific Number</u>	<u>Name</u>	<u>Description</u>	<u>Instrument</u>
SPEX CertiPrep	Calibration Stocks	Catalog Number			ICS 5000/Integrion
	Cl	AS-CL9-2X	1000 mL		ICS 5000/Integrion
	Br	AS-BR9-2X	1000 mL		ICS 5000/Integrion
	NO ₃	AS-NO39-2X	1000 mL		ICS 5000/Integrion
	SO ₄	AS-SO49-2X	1000 mL		ICS 5000/Integrion
High Purity Standards	Secondary Stocks	Catalog Number			ICS 5000/Integrion
	Cl	IC-CL-M	1000 mL		ICS 5000/Integrion
	Br	IC-BR-M	1000 mL		ICS 5000/Integrion
	NO ₃	IC-NO-M	1000 mL		ICS 5000/Integrion
	SO ₄	IC-SS-M	1000 mL		ICS 5000/Integrion

Appendix B. Chromeleon Console view

Console - Easy Access to Data



The screenshot displays the Chromeleon Console interface for 'AS12A Calibration and Samples'. The main window is divided into several sections:

- Navigation Pane (Left):** A tree view showing the project structure, including folders for 'Data', 'Calibration', and 'Samples'. A red box highlights the 'Data' folder.
- Table (Center):** A table listing 10 rows of data. The columns are: #, Name, Type, Position, Volume (µl), Level, and Processing Method. Each row includes a small chromatogram plot.

#	Name	Type	Position	Volume (µl)	Level	Processing Method
1	Seven Inion Standard#1 - 1	Standard	1	25000	01	AS12A Anions.v13.0
2	Seven Inion Standard#1 - 2	Standard	2	25000	02	AS12A Anions.v13.0
3	Seven Inion Standard#1 - 3	Standard	3	25000	03	AS12A Anions.v13.0
4	Seven Inion Standard#1 - 4	Standard	4	25000	04	AS12A Anions.v13.0
5	Seven Inion Standard#1 - 5	Standard	5	25000	05	AS12A Anions.v13.0
6	Seven Inion Standard#1 - 6	Standard	6	25000	06	AS12A Anions.v13.0
7	Blanking/White	Unknown	7	25000		AS12A Anions.v13.0
8	Tap/White	Unknown	8	25000		AS12A Anions.v13.0
9	Power Socket Water	Unknown	9	25000		AS12A Anions.v13.0
10	Power Socket Water	Unknown	10	25000		AS12A Anions.v13.0
- Category Bars (Bottom):** A horizontal bar at the bottom of the console showing various categories like 'Backwards', 'Data', and 'Workflow'. A red box highlights the 'Data' category.

Yellow callout boxes point to the 'Navigation Pane' and 'Category Bars'.

Appendix C. Chromeleon Studio view

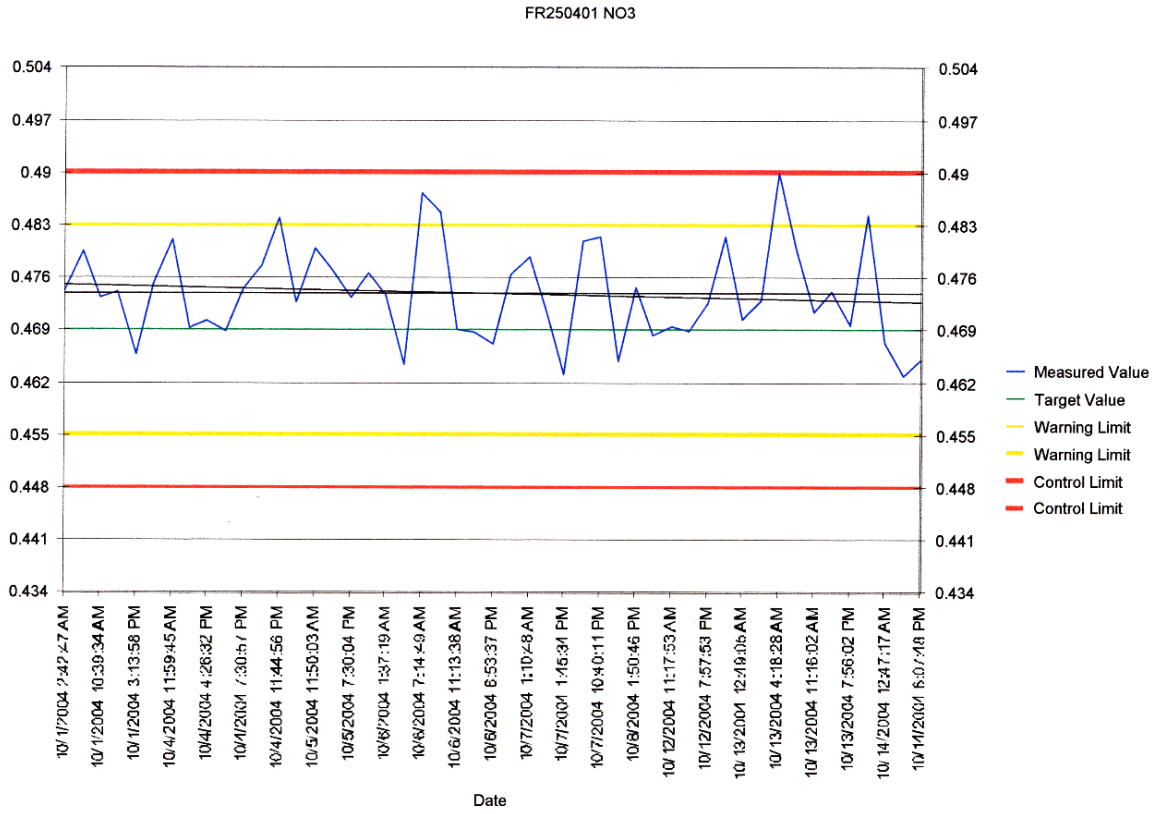
Chromatography Studio - All Details of an Experiment



The screenshot displays the Chromeleon Studio interface. A red box highlights the **Ribbon** at the top, which contains various tool icons for data processing and analysis. On the left, a **Navigation Pane** lists the project's hierarchical structure. Below it, **Category Bars** provide additional context and actions for the selected data. The main window shows a chromatogram with several peaks, and a data table below it.

Peak	Peak Label	Ret. Time (min)	Amount (nmol)	Std. Dev. (nmol)	Area	Height (mV)	Gain	Width (min)	Height (mV)	Area (mV·min)	Peak	Height (mV)
1	Peak 1	1.00	1.00	0.05	10.0	100	1.0	1.0	100	10.0	1	100
2	Peak 2	1.50	1.50	0.05	15.0	150	1.5	1.5	150	15.0	2	150
3	Peak 3	2.00	2.00	0.05	20.0	200	2.0	2.0	200	20.0	3	200
4	Peak 4	2.50	2.50	0.05	25.0	250	2.5	2.5	250	25.0	4	250
5	Peak 5	3.00	3.00	0.05	30.0	300	3.0	3.0	300	30.0	5	300
6	Peak 6	3.50	3.50	0.05	35.0	350	3.5	3.5	350	35.0	6	350
7	Peak 7	4.00	4.00	0.05	40.0	400	4.0	4.0	400	40.0	7	400
8	Peak 8	4.50	4.50	0.05	45.0	450	4.5	4.5	450	45.0	8	450
9	Peak 9	5.00	5.00	0.05	50.0	500	5.0	5.0	500	50.0	9	500
10	Peak 10	5.50	5.50	0.05	55.0	550	5.5	5.5	550	55.0	10	550
11	Peak 11	6.00	6.00	0.05	60.0	600	6.0	6.0	600	60.0	11	600
12	Peak 12	6.50	6.50	0.05	65.0	650	6.5	6.5	650	65.0	12	650
13	Peak 13	7.00	7.00	0.05	70.0	700	7.0	7.0	700	70.0	13	700
14	Peak 14	7.50	7.50	0.05	75.0	750	7.5	7.5	750	75.0	14	750
15	Peak 15	8.00	8.00	0.05	80.0	800	8.0	8.0	800	80.0	15	800
16	Peak 16	8.50	8.50	0.05	85.0	850	8.5	8.5	850	85.0	16	850
17	Peak 17	9.00	9.00	0.05	90.0	900	9.0	9.0	900	90.0	17	900
18	Peak 18	9.50	9.50	0.05	95.0	950	9.5	9.5	950	95.0	18	950
19	Peak 19	10.00	10.00	0.05	100.0	1000	10.0	10.0	1000	100.0	19	1000
20	Peak 20	10.50	10.50	0.05	105.0	1050	10.5	10.5	1050	105.0	20	1050

Appendix D. Control Chart Example












AN.HEAL.IN.IC-anions.13.2

Final Audit Report

2025-01-07

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Document page count:	20
Number of supporting files:	0
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