

Anion Analysis – Chloride and Sulfate

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Purpose: This procedure describes the steps to analyze total chloride and sulfate concentrations in water. The units of total chloride and sulfate for this analysis are milligrams per liter of water.

Sample Holding Time: ≤ 1 year @ 4° C unpreserved

Materials Required:

For analysis/prep:	For standards:
<ul style="list-style-type: none">• Dionex™ ICS 2100 Analyzer• Dionex™ AS-DV Autosampler PolyVials and Caps<ul style="list-style-type: none">○ 5mL PolyVials○ Black 5mL Filter Caps• Acrylic 10-vial rack• Black “hammer” device• Green permanent marker• Red permanent marker• 100/200-1000µL, 5000µL pipettes (<i>for dilutions</i>)• Blue pipette tips (<i>for dilutions</i>)	<ul style="list-style-type: none">• Analytical scale• Fifteen 250 mL plastic Nalgene bottles• MQ water• Clean MQ squirt bottle• 1000ppm Cl and SO₄ stock calibration solutions• 1000 ppm Cl and SO₄ stock ICV solutions (must be different manufacturer than calibration stock solutions)• 1 mL disposable pipettes• 10mL disposable beakers• Paper and pen/pencil

Personal Protective Equipment / Waste Disposal: Nitrile gloves and safety glasses are always required during this procedure. This is not only for your protection, but also to prevent contamination of samples. Always use chemical resistant gloves (not latex).

Quality Assurance/Quality Control:

The validity of the data collected by the instrument is checked with the following quality control samples:

- Reagent blank, plain Milli-Q water
- Initial Calibration Verification (ICV) sample(s) are prepared from a different stock material than the calibration curve.
- Every 10th sample is duplicated.
 - For a full run: 26 samples with 2 duplicates.

Waste Disposal: Excess sample and standard can be disposed of down the drain.

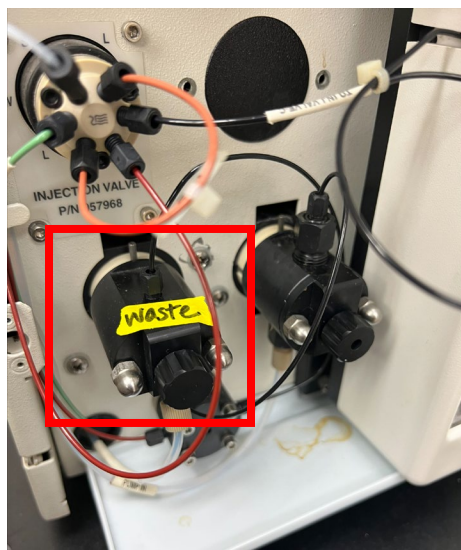
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Autosampler and Instrument Preparation

- 1.0 Take out standards and samples with enough time to warm to room temperature by the time you hit play.
 - 1.1 26 samples for a full run.
- 2.0 Disconnect the instrument from the program by clicking the glowing green button in the ICS-2100 tab.
 - 2.1 See “*Cheat Sheet*” page for a picture reference.
- 3.0 The screen of the instrument (*the control panel*) will display a message (“Suppressor stop for flow rate”) – press **OK**.
- 4.0 Unscrew the eluent cap and dump the MQ water that was left from the last run, and refill with fresh MQ water to the shoulder of the bottle. (Do not top off MQ)
 - 4.1 On the control panel, change the eluent level to 2 liters (L).
- 5.0 Prime the pumps.
 - 5.1 Open the front panel of the ICS.
 - 5.2 Open the pump head on the waste valve (left-hand black knob, labeled waste) halfway (to the left).



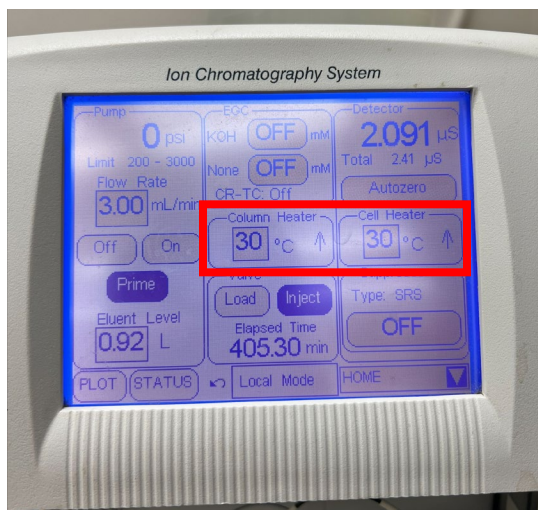
- 5.2.1 (Start turning with your thumb on the top of the valve, and finish turning once your thumb is on the bottom)
- 5.3 Select **Prime** on the control panel and select **OK**.
- 5.4 Let it run for ~10 minutes.
- 5.5 *If the eluent ran dry:*
 - 5.5.1 Insert syringe into the lower right-hand knob and twist one rotation. Gently pull eluent through until no more bubbles appear to go through the tubing (generally four).
 - 5.5.2 You will usually not have to worry about this.

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While the pumps are being primed:

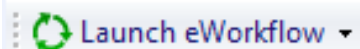
NOTE: Once the ten minutes are up, skip down to steps 16.0-22.0, even if you aren't done with 6.0-15.0, then return to complete 6.0-15.0.

- 6.0 Use the control panel to set the temperature of the column and cell heaters to 30°C.

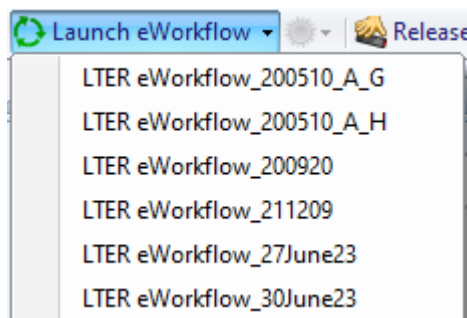


- 7.0 Create new eWorkflow by:

- 7.1 Selecting **Launch eWorkflow**



- 7.2 In the dropdown menu, select the workflow that has the most recent date.



- 8.0 In the window that pops up, type in your number of samples (28 for a full run).

- 8.1 If you click **Sample Start Position** the sample rack displayed on screen will fill.

- 8.2 Click **Next**.

- 9.0 Name your sequence:

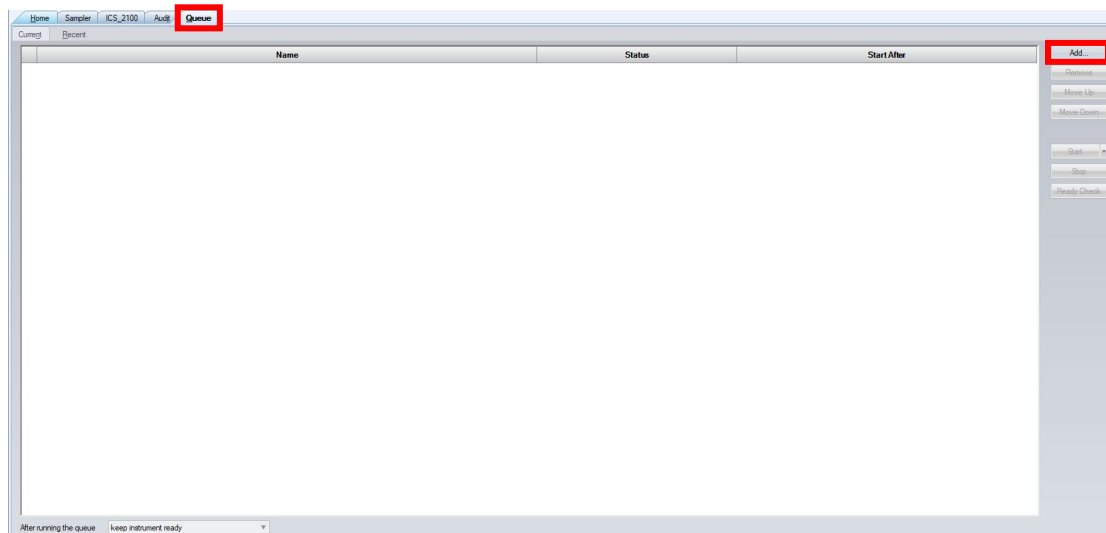
- 9.1 Should be labeled "month_day_year_ANIONS_project(s)"

9.1.1 Ex) 07_14_2023_ANIONS_LTER

9.1.2 Ex) 07_15_2023_ANIONS_SaltyP_Ledin

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- 9.2 Leave a comment if something in the instrument was changed (ex: the column), or if there was a split run.
- 9.3 Sequence Location: Instrument Data → ICS_2100 → (year)
- 9.4 Click **Finish**
 - 9.4.1 This may take up to a minute to load.
- 10.0 Add the eWorkflow you just created to the queue.
- 10.1 Go to the “Queue” tab towards the top of the window.



- 10.2 Click **Add** (located on the left of the Queue window) and select the eWorkflow you created in the files.
- 11.0 Double click the eWorkflow in the queue to open the “Studio” window.
- 12.0 In the “Studio” window, right click your selected processing method, and click **Assign to Injection**.
 - 12.1 The grayed-out boxes next to the standards should now have a dropdown menu.
 - 12.2 Set the levels of calibration standards.
 - 12.2.1 A=1, B=2, C=3, etc.
 - 12.2.2 Make sure to do this for the check standards too (the last three at the end of the run)
- 13.0 Save the run by clicking the floppy disk icon in the upper left corner.
 - 13.1 A window will pop up. Click **Save**.
- 14.0 Scan in your samples.
 - 14.1 Highlight the first blank sample (position 19) and scan. Hit the down arrow after every scan.

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LTER_ANIONS_7_11_2023_BouncyPonds_SaltyP																	
Queue#	ECID	Name	Type	Level	Position	Volume	Processing Method	Instrument Method	Status	Inject Time	Weight	Dilution	InStd	Replicate ID	Spike Group	Comment	GUID
1	None	MG	Unknown	7	7	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
2	None	STD BLK	Unknown	2	2	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
3	None	STD A	Calibration Standard	3	3	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
4	None	STD B	Calibration Standard	4	4	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
5	None	STD C	Calibration Standard	5	5	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
6	None	STD D	Calibration Standard	6	6	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
7	None	STD E	Calibration Standard	7	7	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
8	None	STD F	Calibration Standard	8	8	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
9	None	STD G	Calibration Standard	9	9	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
10	None	STD H	Calibration Standard	10	10	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
11	None	STD I	Calibration Standard	11	11	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
12	None	STD J	Calibration Standard	12	12	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
13	None	MG	Unknown	13	13	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
14	None	LOG	Unknown	14	14	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
15	None	ICV L	Unknown	15	15	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
16	None	ICV M	Unknown	16	16	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
17	None	ICV H	Unknown	17	17	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
18	None	MG	Unknown	18	18	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
19	None	Sample	Unknown	19	19	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
20	None	Sample	Unknown	20	20	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
21	None	Sample	Unknown	21	21	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
22	None	Sample	Unknown	22	22	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
23	None	Sample	Unknown	23	23	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
24	None	Sample	Unknown	24	24	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
25	None	Sample	Unknown	25	25	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
26	None	Sample	Unknown	26	26	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
27	None	Sample	Unknown	27	27	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
28	None	Sample	Unknown	28	28	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
29	None	Sample	Unknown	29	29	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
30	None	Sample	Unknown	30	30	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
31	None	Sample	Unknown	31	31	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
32	None	Sample	Unknown	32	32	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
33	None	Sample	Unknown	33	33	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
34	None	Sample	Unknown	34	34	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
35	None	Sample	Unknown	35	35	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
36	None	Sample	Unknown	36	36	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
37	None	Sample	Unknown	37	37	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
38	None	Sample	Unknown	38	38	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
39	None	Sample	Unknown	39	39	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
40	None	Sample	Unknown	40	40	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				
41	None	Sample	Unknown	41	41	5000		LTER instrument method	Idle		1.0000	1.0000	1.0000				

14.2 Duplicate every 10 samples.

14.2.1 26 samples with two duplicates for a full run.

15.0 Save again.

Once the 10 minutes are up and the pumps have been fully primed:

16.0 Turn off the pump and tighten the waste valve.

17.0 Connect the instrument again by going back to the “Console” window (in the ICS_2100 tab) and selecting the green button.

18.0 Turn on the pump.

18.1 You should hear a click.

18.2 Watch the bubbles move once you turn on the pump.

18.3 Once the pump is on the pressure will start to climb. Target pressure is above 2000, but if the pressure is staying consistent, you should be ok to run.

18.3.1 If pressure is not climbing, try tightening the waste valve (you may have not tightened it enough).

19.0 Turn on the eluent generator.

19.1 Type 20 µmoles into the textbox and click **On**.

19.2 See “Cheat Sheet” page for a picture reference.

20.0 Turn on the CR-CT by sliding the slider to the left.

21.0 Wait ~5 minutes to turn on the suppressor, to make sure the eluent can get there.

21.1 Make sure there is a full slug of water (no bubbles) before you turn on the suppressor.

21.1.1 Bubbles are okay once you turn the suppressor on (it means it’s working!).

21.2 Set the current to 50 mA.

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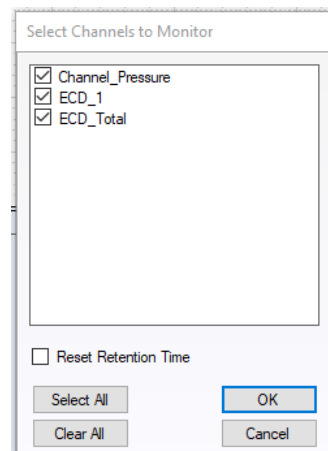
22.0 Monitor the pressure and conductivity baseline by pressing the button on the top of the window that looks like a squiggly graph on a computer screen.



22.1 A new window will open. Make sure all three boxes are checked – do not check **Reset Retention Time** – and press **OK**. (as shown below)

22.2 Autozeroing the conductivity can give you a better idea of the change the conductivity has undergone in a set amount of time.

22.3 We are looking for a stable pressure (ideally above 2000) and a conductivity below 0.5 μS .



23.0 Remove the vials from the previous run.

23.1 Press the carousel button on the left of the autosampler to be able to access the vials under the sampler.

23.1.1 This will take a moment as the autosampler removes itself from the last vial from your previous run.

24.0 Go back to the “Console” window and check the baselines.

24.1 If they haven’t reached ideal conditions (listed above), continue to monitor them.

25.0 Pour your samples and standards.

25.1 Label the plastic PolyVial with the standard type/the sample ID.

25.2 Put the PolyVial into the acrylic vial rack and put paper towel underneath.

25.3 Pour the sample/standard above the line on the PolyVial.

25.3.1 The line is very faint. If your vials are in the acrylic vial rack, pour the sample/standard above where the rack supports the vial.

25.3.2 It is better to overfill than underfill.

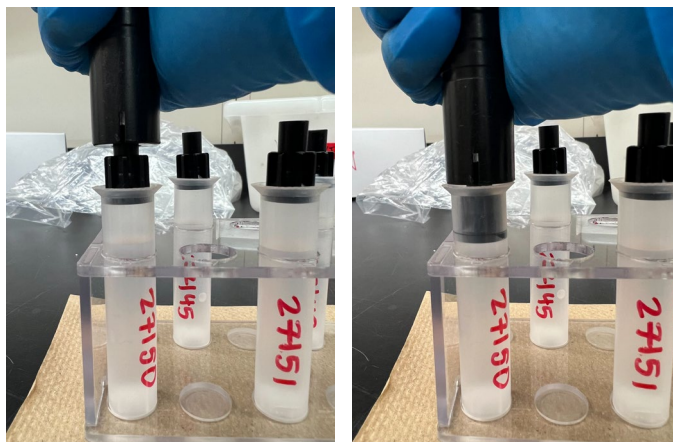


25.4 Put the black filter cap on with the larger side down.

25.5 Use the black “hammer” device to fully insert the cap.

25.5.1 Use the side with the hole first, then the flat side so that the top of the filter cap is about in line with the top of the vial. *The slower you do this the less will splatter.*

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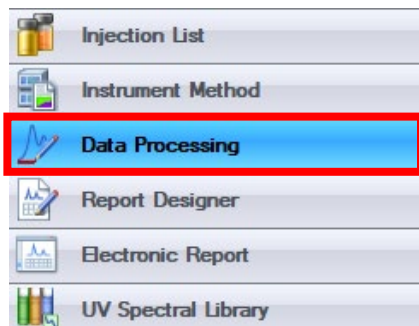


- 25.5.2** Make sure to wipe up any sample or standard that splatters to avoid any cross contamination. You can use the paper towel the acrylic rack is on top of to do this.
- 25.6** While holding onto the vial, whip it towards a sink to get rid of the excess water that is on top of the filter cap.
- 25.7** Use the sample table you made in the “Studio” window to make sure that the vials are going into the right spot.
- 25.8** Put a green dot on top of sample vials that have been poured and “DUP” on top of bottles that are being duplicated.
- 25.9** You can leave the 50th spot (labeled “SHUTDOWN”) empty.
- 26.0** Once all the samples are in, press the carousel button again to lock it in.
- 27.0** Close the hood of the auto sampler.
- 28.0** When you’re ready to hit play, fill out the logbook.
- 28.1** For a full run (28 samples), runtime is ~14 hours.
- 28.2** In the comments section, write: STD A retention time (which you’ll find after the run is complete), the project, and the Sample ID range of what samples you’re running.
- 29.0** In the “Queue” tab of the “Console” window click **Start**.
- 29.1** A window will pop up, select **Stop Monitoring Baseline** and it will start up.

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Data Processing

- 1.0 Once your run is complete, select the “Data Processing” tab on the bottom left in the “Studio” window.



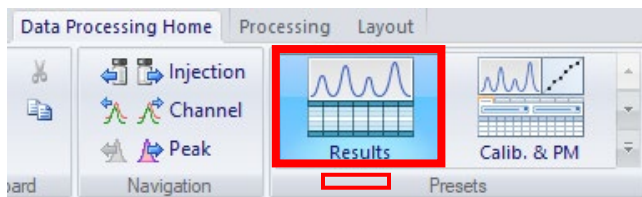
- 2.0 Adjust peak retention time if needed.

- 2.1 Check that the number given in the table is approximately the same as what is shown on the graph.

- 3.0 Select **Calib. & PM**, then in the “Processing” tab (located at the top of the window), select **Cobra Wizard** and go through the prompts.



- 4.0 After going through Cobra Wizard, select **Results** in the “Data Processing Home” tab and select the “Calibration” tab on the bottom.



Peak No.	Peak Name	Ret. Time (min)	Cal. Type	Eval. Type	Number of Points	Rel. Std. Dev. (%)	Coeff. of Determination	C0 (Offset)	C1 (Slope)	C2 (Slope)
4	Chloride	5.664	Lin. With Offset	Area	10	0.6904	0.99996	-0.1526	0.2562	0.0000
5	Sulfate	10.387	Lin. With Offset	Area	8	2.0585	0.99972	-0.1872	0.1949	0.0000
6	Maximum					2.0585	0.99996			
7	Minimum					0.6904	0.99972			

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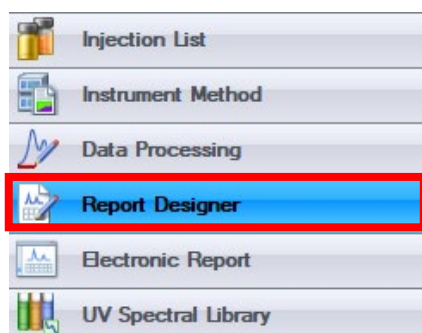
5.0 In the Calibration tab, check the r^2 values.

5.1 Ideally shooting for above 0.999.

5.2 Enter the values into the **IC QC Log**.

5.2.1 *Located: LTER Water Chem Lab → 1 - SOPs and Project Descriptions → 3 - Logs and Data Sheets*

6.0 Go to the “Report Designer” tab in the “Studio” window.



6.1 Then select the “Cl and SO4” tab on the bottom of the screen

	A	B	C	D	E	F	G
1							
2	CHLORIDE						
3	Chloride	Name	Retention Time	Area	Height	Amount	
4			min	$\mu\text{S}^2/\text{min}$	μS	mg/L	
5			ECD, 1	ECD, 1	ECD, 1	ECD, 1	
6			Chloride	Chloride	Chloride	Chloride	
7	1	MO	5.630	0.0011	0.00	0.5777	
8	2	STD BLK	n.a.	n.a.	n.a.	n.a.	
9	3	STD A	5.664	0.4476	2.36	2.2549	
10	4	STD B	5.667	0.9461	4.66	4.1280	
11	5	STD C	5.667	1.4534	7.35	6.0337	
12	6	STD D	5.670	1.9910	9.95	8.6336	
13	7	STD E	5.671	2.5146	12.45	10.0207	
14	8	STD F	5.674	5.1684	25.18	19.9907	
15	9	STD G	5.674	10.3791	51.11	39.9955	
16	10	STD H	5.681	15.7493	77.59	59.7418	
17	11	STD I	5.687	21.0146	102.51	79.5231	
18	12	STD J	5.691	26.5362	126.62	100.2670	
19	13	MO	5.667	0.0024	0.01	0.4526	
20	14	LOD	5.660	0.0068	0.03	0.5992	
21	15	ICV L	5.667	0.1979	1.08	1.3189	
22	16	ICV H	5.670	1.6468	8.52	6.7659	
23	17	ICV H	5.671	7.5013	37.19	28.7549	
24	18	MO	n.a.	n.a.	n.a.	n.a.	
25	19	Snowpile top 520	5.607	190.9559	797.96	718.0122	
26	20	Snowpile md 526	5.607	196.5947	895.23	735.4021	
27	21	Snowpile btm 526	5.617	211.1722	858.43	793.9251	
28	22	Snowpile top 109	5.620	211.8790	869.88	796.5895	
29	23	Snowpile md 106	5.621	210.9699	869.47	793.1953	
30	24	Snowpile btm 109	5.620	216.2710	876.43	813.0807	
31	25	Snowpile top 116	5.763	122.0831	534.86	459.2268	
32	26	Snowpile md 116	5.760	122.2774	534.74	459.9569	
33	27	Snowpile btm 116	5.767	129.7643	564.14	488.0644	
34	28	Snowpile top 1120	5.771	136.7169	589.74	514.2047	
35	29	Snowpile top 1120 DUP	5.767	136.6716	588.21	514.0344	
36	30	Snowpile md 1120	5.767	136.3034	587.54	512.9517	
37	31	Snowpile btm 1120	5.767	136.6336	588.62	513.8917	
38	32	GW Snowpile top 124	5.750	112.8552	495.64	424.5884	
39	33	GW Snowpile md 124	5.750	112.9663	496.27	425.0563	
40	34	GW Snowpile btm 124	5.757	123.7542	538.58	465.5048	
41	35	Spring L1	5.667	0.3040	1.65	1.7155	
42	36	Spring L7	5.671	13.8689	69.66	53.1263	
43	37	Spring L13	5.667	26.5587	130.19	100.3519	
44	38	Spring L19	5.767	127.9118	556.61	481.1240	
45	39	Spring O1	5.667	0.3390	1.84	1.8470	
46	40	Spring O1 DUP	5.667	0.3279	1.79	1.8053	
47	41	Spring O7	5.677	12.9283	64.42	49.1439	
48	42	Spring O13	5.667	26.1519	128.52	98.8235	
49	43	Spring O19	5.777	147.0577	631.51	552.8604	
50	44	Spring O1	5.667	0.4024	3.63	3.4193	

7.0 Check and compare the ICVs to predicted values by entering them into the **IC QC Log**.

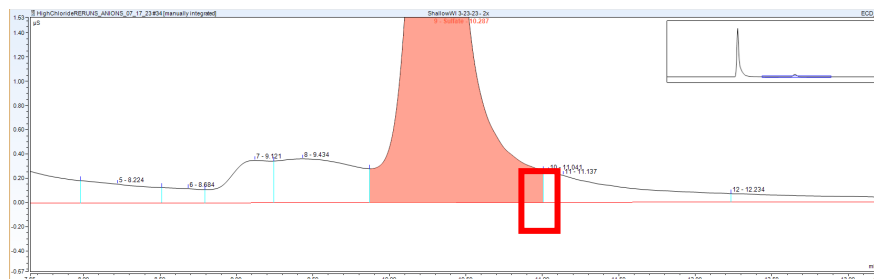
7.1 Also glance at standard values to see if they are similar to actual concentrations.

8.0 Check that duplicates are similar for both chloride and sulfate. If they’re not, go back to data processing and check the integration. It is likely that one curve has a longer tail than the other.

8.1 To fix this, manually integrate one of the curves to end at approximately the same time as the other.

8.1.1 To do this place your cursor over the teal line and move it.

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8.1.2 If the baseline tilts when you move the integration window, make sure to straighten it by hovering over the small blue line (your cursor should turn into a sideways T-shape) at the end of the window and moving your mouse up/down.

9.0 Export the data:

9.1 Select the entire report (like you would in a spreadsheet).

9.2 Copy and paste into an excel sheet.

9.3 Name the excel sheet the same as your sequence.

9.3.1 *Ex) LTER_ANIONS_07_07_2023*

9.4 Save the excel sheet into the anions raw data folder.

9.4.1 *LTER Water Chem Lab → RAW DATA → Anions → (year)*

10.0 Highlight any values above 100mg/L.

10.1 *(See next page for dilution instructions)*

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Dilutions

If you are obtaining anion concentrations above the standard curve, you will need to preform dilutions and rerun the sample so the concentration lie within the curve.

- 1.0 Note all the samples in your runs that are over 100mg/L for chloride, making sure to note the concentration values.
- 2.0 Perform the original procedure as usual, until you get to the sample preparation step (Step 25.0).
- 3.0 Using a pipette, prep samples according to the values you noted and the table below:

Initial Chloride (mg/L)	Sample Volume (mL)	MQ Volume (mL)	Dilution Factor
100-150 mg/L	2.5	2.5	2
151-250 mg/L	2	3	3
251-400 mg/L	1	4	5
401-900 mg/L	0.5	4.5	10
901-1800mg/L	0.25	4.75	20

(1000 μ L = 1mL)

- 4.0 In the Sample Table in the Studio window, add the dilution factor after the sample ID.
Ex) 123454 - 10x

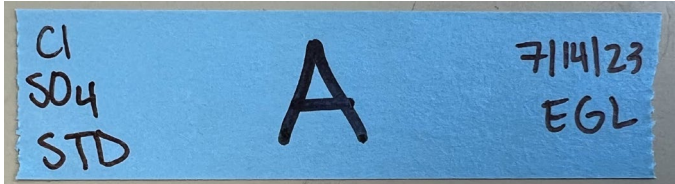
NOTE: The total sample volume will be below the line of the PolyVial that you would usually fill the sample to/above. This is OK.

- 5.0 Place filter caps on according to the original procedure, but in addition, use a smaller tool to insert the cap to make sure the water flushes through the cap.
 - 5.1 Before fully inserting the cap, invert PolyVials to mix sample and Milli-Q.
- 6.0 Continue with your procedure as usual.

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Preparing Reagents

Calibration Standards and ICVs

- 1.0 Go to standard calc sheet folder in share drive.
 - 1.1 *LTER Water Chem Lab* → *SOPs and Project Descriptions* → *STANDARD CALC SHEETS* → *Cl-SO4*
- 2.0 Make a copy of the file titled **CI_SO4-TEMPLATE_Blank**
- 3.0 Rename the file with the date that you plan to make the standards.
 - 3.1 *Ex) CI_SO4_07_14_2023*
 - 3.2 Put the file in the file titled with the appropriate year.
- 4.0 Open the sheet and fill out your name and the date of preparation in the top right corner of the sheet, as well as the stock solution information.
- 5.0 Print a copy of the calculation sheet.
- 6.0 Obtain fifteen clean 250 mL plastic Nalgene bottles.
 - 6.1 Brand new bottles are ideal, but freshly acid washed bottles are also okay. (See acid washing SOP)
- 7.0 Label the bottles with label tape:
 - 7.1 A, B, C, D, E, F, G, H, I, J, Blk, LOQ, ICV-L, ICV-M, ICV-H
 - 7.2 On the left side of the tape write “Cl SO4 STD” and on the right side write the day of preparation and your initials
 - 7.3 *Ex)* 
- 7.4 *Tip: color coding the calibration standards vs. ICVs may also be helpful*
 - 7.4.1 *Ex) all calibration standards (A-J and Blk) are blue and all ICVs (LOQ, ICV-L, M, H) are pink.*
- 8.0 Use the file titled **CI_SO4-TEMPLATE** (located in the Cl-SO4 standard calc sheet folder) to find the concentrations of the stock solution you must use to make the standard and ICV solutions.
 - 8.1 There should be a copy of it near the analytical scale in the River Ecology lab. If not, print one out to reference.
- 9.0 Use an analytical scale and the template to make the standards and ICVs:

NOTE: The amount of the stock solution and MQ water on the calculation sheet is cumulative.

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Example: For standard A, add 0.500g of Cl^- stock, and then add 0.500g of SO_4^{2-} stock (so that the total weight is now 1.000g). Then add 249g of MQ water (so that the total weight is now 250.00g).

$\text{Cl}^- / \text{SO}_4^{2-}$ IC STANDARDS				DATE:	
Cl^- & SO_4^{2-} stocks are 1,000 ppm (1,000,000 ppb)				Prepared By:	
				Cl^-	SO_4^{2-}
Calibration Standards	Brand of Stock				
	Lot #				
ICV Standards	Brand of Stock				
	Lot #				
Standard	Cl^- Stock (g)	SO_4^{2-} Stock (g)	MQ (g)		
A	0.500	1.000	250.00	A	2.00 2.00
B	1.000	2.000	250.00	B	4.00 4.00
C	1.500	3.000	250.00	C	6.00 6.00
D	2.000	4.000	250.00	D	8.00 8.00
E	2.500	5.000	250.00	E	10.00 10.00
F	5.000	10.000	250.00	F	20.00 20.00
G	10.000	20.000	250.00	G	40.00 40.00
H	15.000	30.000	250.00	H	60.00 60.00
I	20.000	0.000	250.00	I	80.00 ----
J	25.000	0.000	250.00	J	100.00 ----
Blk	0.000	0.000	250.00	Blk	0.00 0.00
LOQ	0.010	0.025	250.00	LOQ	0.04 0.06
ICV	Cl^- Stock (g)	SO_4^{2-} Stock (g)	MQ (g)		Cl^- (mg/L) SO_4^{2-} (mg/L)
L	0.250	0.500	250.00	L	1.00 1.00
M	1.750	3.500	250.00	M	7.00 7.00
H	7.500	15.000	250.00	H	30.00 30.00

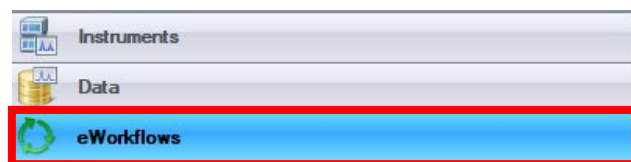
NOTE: use ICV standard stock solution to make the LOQ

- 9.1 Place a 250mL Nalgene bottle on the scale, close all the doors on the scale, and tare.
- 9.2 Pour the stock solution into a disposable 10mL beaker.
 - 9.2.1 This is to limit contamination of the stock solution and also helps you have more control over the amount you pour.
- 9.3 For low-end amounts of stock solution use the 1mL disposable pipette.
 - 9.3.1 One droplet is ~0.01g
- 9.4 For high-range amounts of stock you can pour the stock straight from the bottle, until you get close to the desired weight, then switch back to using the disposable pipettes and beakers.
- 9.5 Write down the exact amount of stock solution on the blank calculation sheet you printed out, making sure to close the doors of the analytical scale before recording your measurement.
- 9.6 To add MQ:

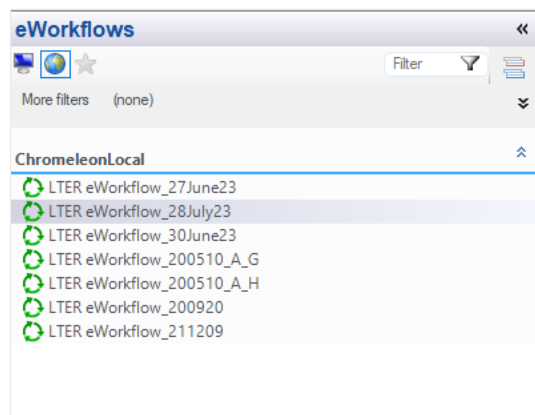
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- 9.6.1** Take the cap off the MQ squirt bottle, take the standard/ICV bottle off the scale, and pour water to about 0.5cm below the shoulder of the standard/ICV bottle.
- 9.6.2** Then, put the cap back on the MQ squirt bottle, return the standard bottle to the scale and squirt water into the bottle until you reach 250mL.
- 10.0** Once you have made all of your standards, put them in the bin labeled **ANION STDs** and put in the refrigerator in the River Ecology lab.
- 10.1** Dump old standards if needed.
- 11.0** Enter in your stock solution amounts into your standard calculation sheet on a computer to find the concentration values.
- 12.0** Print out your newly filled out sheet.
- 13.0** Add this to the **IC Chromatography SOP and Log** binder that sits next to the IC computer.
- 14.0** Add a new eWorkflow:

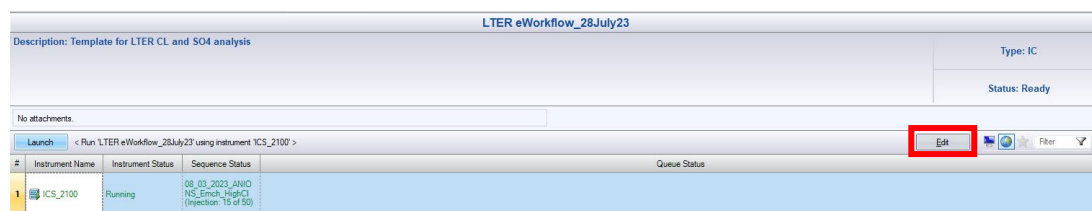
- 14.1** Go to the “Console” window, and select the eWorkflows tab on the bottom left:



- 14.2** Select the most recent eWorkflow in the list on the left of the window.



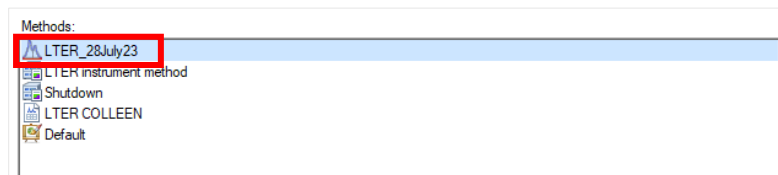
- 14.3** Then select edit.



- 14.4** This will open the “eWorkflow Editor” window.

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- 14.5 In the “eWorkflow Editor” window, right click the processing method in the “Methods” box and select **Open**.



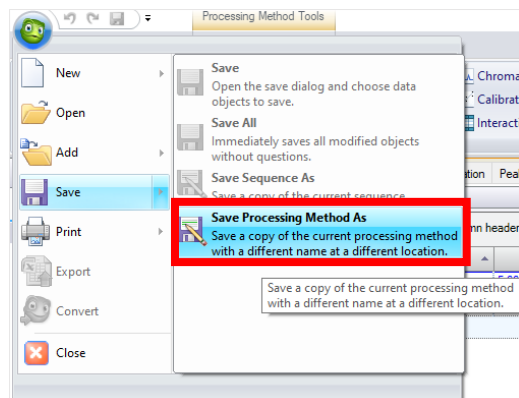
- 14.6 This will open a new window.

- 14.7 In this window, change the levels of your calibrants in the component table reflecting the calculated values of your standard prep.

Group Area	Name	Ret Time	Window	Channel	Eval Type	Stand Meth	Cal Type	Level '01'	Level '02'	Level '03'	Level '04'	Level '05'	Level '06'	Level '07'	Level '08'	Level '09'	Level '10'
Chloride	5.354	5.000	RF	All Channels	Area	External	Lin, Wt%Offset	2.000000	4.000000	6.010000	8.000000	10.040000	20.000000	40.000000	58.900000	79.450000	100.00
	10.444	5.000	RF	All Channels	Area	External	Lin, Wt%Offset	2.000000	4.040000	6.010000	8.990000	10.060000	20.030000	39.990000	58.930000		

- 14.7.1 1 = Standard A, 2 = Standard B, 3 = Standard C, etc.

- 14.8 Click on Chromeleon in the top left corner of the window and select **Save**, then **Save Processing Method As**.



- 14.8.1 Location: *Instrument Data* → *Processing Methods*

- 14.8.2 Name the new processing method: *LTER_date*

Ex) *LTER_27July23*

- 14.9 Go back to “eWorkflow Editor” window and remove the old processing method from the methods box (by left clicking the method and clicking **Remove**).

- 14.10 Add the new processing method you just made by selecting **Add**.

- 14.11 Select **File** (in the top left corner) and **Save As**.

- 14.11.1 Name the new eWorkflow: *LTER eWorkflow_date*.

Ex) *LTER eWorkflow_27July23*

NOTE: Make sure the date on the standard bottles, standard calc sheet, processing method, and eWorkflow are all the same.

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Cheat Sheet

The screenshot displays the control interface for an ICS-2100 instrument. The interface is organized into several sections with various controls and readouts:

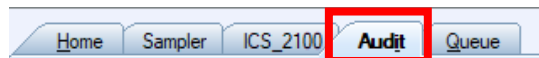
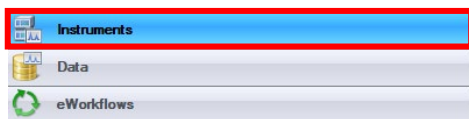
- Step 2.0 & 17.0:** A green indicator light is labeled "Connected". A "Continue" button is visible to the right.
- Step 18.0:** The "Pump" section includes "On" and "Off" buttons. The "Flow" rate is set to 1.00 [ml/min] and the "Pressure" is 2008.
- Step 21.0:** A "Prime" button is located below the pump controls. The "Eluent Fill Level" is shown as a horizontal bar with a diamond marker at the 2 position on a scale from 0 to 4.
- Step 19.0:** The "Valves" section features a circular diagram with labels (S), (L), (C), (P), (L), and (P). The "InjectPosition" is set to (C).
- Step 20.0:** The "Eluent Generator" section has "On" and "Off" buttons, with "Concentration" set to 20.00 [mM]. The "EGC % Remaining" is 40.7% and "CR-TC" is set to "On".
- Step 21.0:** The "Suppressor" section shows the "Type" as AERS_4mm and "Current" as 50 [mA]. The "Column Heater" section has a "Set Temperature" of 30.0 [°C] and a "Measured Temperature" of 29.9 [°C].
- Conductivity:** The "Autozero" button is present. The "Total" conductivity is 0.753 [μS].

ICS-2100

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Hot Tips!

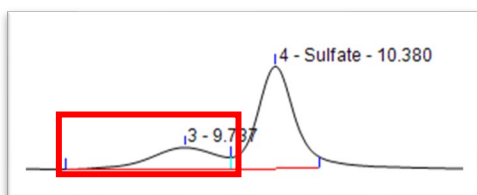
1. If you forgot to turn on the heat baths, the pressure will likely be around 2245psi, as opposed to 2045psi.
2. If you forgot to fill out the log book at the start of your run, go to the **Audit** tab (located in the Console window, under the Instrument tab)



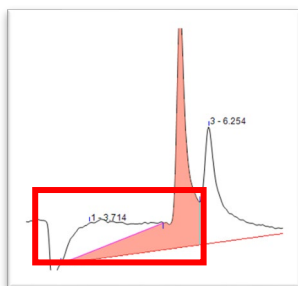
Look for the date and time the run started - the pressure and total signal should be listed.

23	8/2/2023	11:46:22 AM -05:00	0.500	ICS_2100	Log Background: 0.33 [µS]
24	8/2/2023	11:46:22 AM -05:00	0.500	ICS_2100	Log Pressure: 2031.47 [psi]

3. Double peaks in sulfate may be from other contamination like nitrate.



← If you're having trouble isolating the sulfate (second, larger) peak from the contamination (first, smaller) peak



← Note: This picture is from a chloride peak, but you may see this same issue in sulfate peaks that contain contamination.*

Right click the contamination peak and delete it. Then use the split peak function (processing tab when Calib. & PM is selected) so that the integration only includes the sulfate peak.



4. If you get this message after you try to start the run (pictured below), just select **Start** again and the run will start.

