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Section: 01

TA: Supriya

Ion Chromatography – Anions in Seawater

	GRADING	Max	Points
1	Abstract		
2	Table summarizing important instrumental parameters with Instrumental set-up		
3	Calibration factor for pipette		
4	Table presenting retention times and instrumental sensitivity for single anion with ordered list of sensitivity for different anions		
5	Calibration curves for anions		
6	Table presenting best fit of calibration curves		
7	Chromatogram of sea-water sample		
8	Calculation of the concentration of each identified anion in seawater		
9	Chromatogram of Unknown		
10	Calculation of the concentration of the anion in the Unknown		
11	Table of comparison with gravimetric analysis		
12	Comparison with LEO-15		
13	Answer to questions		

Abstract:

Through the experiment, the collected data yielded the calculations of the concentrations of the anions in the unknown sample to be 4.12626105ppm for Fluoride, 6.023264385ppm for Chloride, 23.9117666ppm for Phosphate, and 20.95579338ppm for Sulfate. Within the seawater samples, the average concentrations found within the two samples were 17244.85928ppm for Chloride and 2056.49385ppm for Sulfate.

Introduction:

Understanding the concentration of each anion within the seawater is important to individuals to better understand seawater. The amount of research that can be done with the known concentrations of anions is gigantic. This information further allows individuals to be cautious in what the individuals do and how the individuals can filter the seawater to prepare the future of the environment for any crisis.

Experimental Methods:

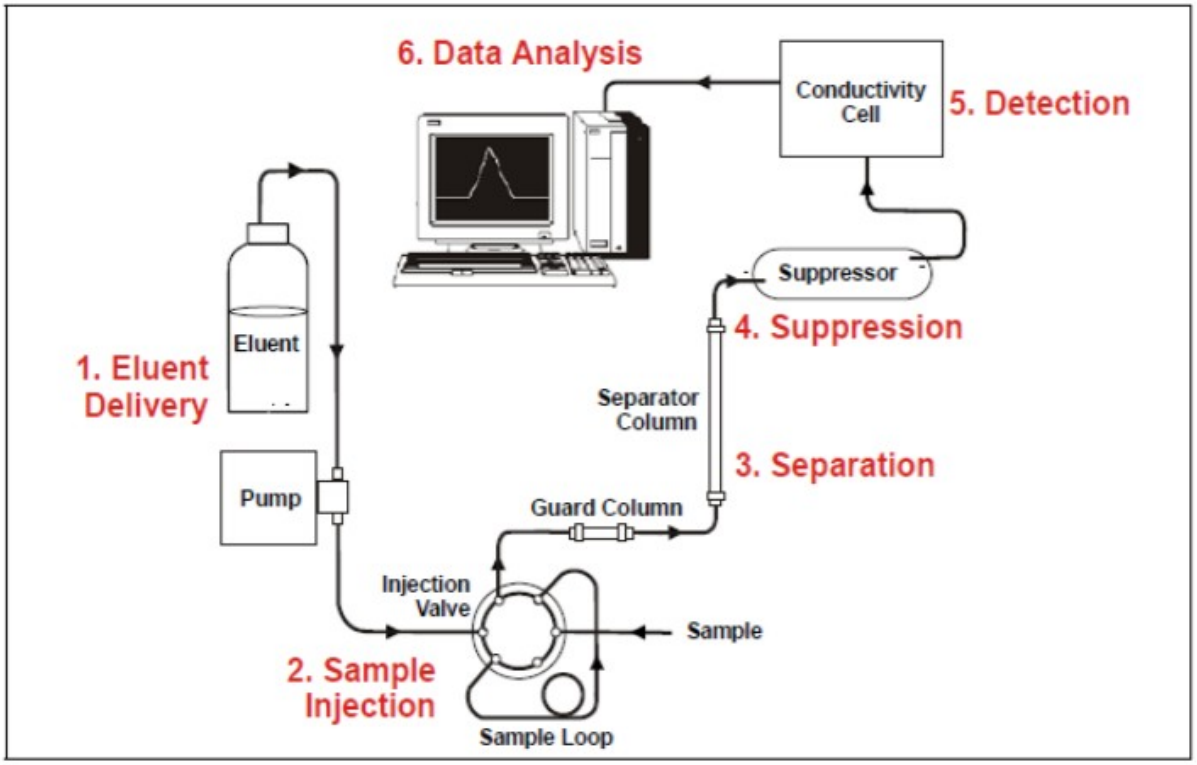

Table I: Various numerical components of Schroeder Machine:

Flow Rate	1.20 microL/min
Pump Pressure	~1500PSI
Eluent	HCO3- and CO32-

Table I contains numerical values for machine flow rate, pump pressure, and the eluent.

The process this machine undergoes is first through the injection of the sample. After the injection of the sample, eluent made up of HCO3- and CO32- goes through with the first guard column then goes through the separator column where it gets prepared to get pushed through the suppressor. The suppressor suppresses the conductivity of the sample and goes through the conductivity cell which leads to the machine giving a data analysis. Through that process, after the suppression, the suppressor has to regenerate using the excess eluent that is there.

Results and Discussion:

Table II: Raw Collected Data

NaF mass:	.1106g
NaCl mass:	.082g
Na2SO4 mass:	.0736g
NaH2PO4 2H2O mass:	.0346g
Pipette Calibration:	4.9995mL per 5mL
ACPS Prepared Solution	
NaF mass:	.0884g
NaCl mass:	.0991g
Na2SO4 mass:	.3713g
NaH2PO4 2H2O mass:	.4348g

Table I contains the data for student prepared solutions, pipette calibration to deliver the seawater, and the stockroom prepared ACPS solution.

Table III: Prepared Standards' Calculated Data

	Area (microS*min)	Concentration (ppm)	Sensitivity (microS*min/ppm)	Retention Time (min)
Chloride	187.053	49.75	3.7599	4.007
Fluoride	132.804	50.02	2.655	2.670
Sulfate	118.593	49.77	2.3828	9.533
Phosphate	23.815	21.063	1.1307	7.497

Table III includes the area, concentration, sensitivity and retention times with the anions ranked from most sensitive to least sensitive.

Calculations:

Fluoride: Area: 278.416 x .477 = 132.804 microS*min

Concentration: .1106g x 1 mol NaF / 41.99g NaF x 1 mol F- / 1 mol NaF x 18.99g F- x 1000mg / 1g = 50.02ppm

Sensitivity: 132.804 / 50.02 = 2.655 microS*min/ppm

Chloride: Area: 473.553 x .395 = 187.053 microS*min

Concentration: .082g x 1mol NaCl / 58.44g NaCl x 1 mol Cl- / 1 mol NaCl x 35.453g Cl- x 1000mg / 1g = 49.75ppm

Sensitivity: 187.053 / 49.75 = 3.7599 microS*min/ppm

Phosphate: Area: 46.154 x .516 = 23.815 microS*min

Concentration: .0346g x 1 / 156.0076g x 1 / 1 x 94.9714g x 1000mg / 1g = 21.063 ppm

Sensitivity: 23.815 / 21.063 = 1.1307 microS*min/ppm

Sulfate: Area: 132.506 x .895 = 118.593 microS*min

Concentration: .0736g x 1/142.04g x 1 / 1 x 96.06g x 1000mg / 1g = 49.77ppm

Sensitivity: 118.593 / 49.77 = 2.3828 microS*min/ppm

Table IV: ACPS, Unknown, and Seawater Samples

Fluoride	Concentration (ppm)	Height(µS)	Width(min)	Area (µS*min)	Retention Time (min)
ACPS 10-fold	2	4.564	0.178	0.812392	2.44
ACPS 4-fold	5	11.389	0.162	1.845018	2.44
ACPS undiluted	20	45.077	0.171	7.708167	2.457
Chloride	Concentration (ppm)	Height(µS)	Width(min)	Area (µS*min)	Retention Time (min)
ACPS 10-fold	3.005	3.008	0.214	0.643712	3.833
ACPS 4-fold	7.5125	8.103	0.21	1.70163	3.827
ACPS undiluted	30.05	39.408	0.205	8.07864	3.837
Phosphate	Concentration (ppm)	Height(µS)	Width(min)	Area (µS*min)	Retention Time (min)
ACPS 10-fold	14.963	2.267	0.462	1.047354	7.633
ACPS 4-fold	37.4075	6.298	0.455	2.86559	7.593
ACPS undiluted	149.63	29.592	0.479	14.174568	7.453
Sulfate	Concentration (ppm)	Height(µS)	Width(min)	Area (µS*min)	Retention Time (min)
ACPS 10-fold	12.56	3.282	0.598	1.962636	10.057
ACPS 4-fold	31.4	9.004	0.591	5.321364	10.017
ACPS undiluted	125.6	40.704	0.632	25.724928	9.853
Unknown	Concentration	Height(µS	Width(min	Area	Retention

	(ppm)	)	)	(µS*min)	Time (min)	
Fluoride	4.12626105	9.56	0.166	1.58696	2.44	
Chloride	6.023264385	7.182	0.223	1.601586	3.823	
Phosphate	23.9117666	4.887	0.457	2.233359	7.607	
Sulfate	20.95579338	7.149	0.593	4.239357	10.037	
Seawater 1	Concentration	Height(µS)	Width(min)	Area (µS*min)	Retention Time (min)	Original Concentration (ppm)
Fluoride	None					
Chloride	16.95235051	22.315	0.202	4.50763	3.823	16955.74166
Phosphate	None					
Sulfate	2.034602076	0.686	0.6	0.4116	10.09	2035.009078
Seawater 2	Concentration	Height(µS)	Width(min)	Area (µS*min)	Retention Time (min)	Original Concentration (ppm)
Fluoride	None					
Chloride	17.5304701	23.076	0.202	4.661352	3.817	17533.9769
Phosphate	None					
Sulfate	2.077563025	0.697	0.603	0.420291	10.103	2077.978621

Table IV contains concentration, height, width, area of peak, retention time for all ACPS, unknown, and seawater samples.

Calculations:

Pipette Calibration:

4.9995mL / 5mL = .9999 / 100 x ((.999)x10) / 100 = .0009998 calibrated factor

ACPS Calibrations:

NaF: .0884 g NaF / 2L x 1 mol NaF / 41.99g NaF x 1 mol F- / 1 mol NaF x 18.99g F- x 1000mg / 1g = 20.0ppm

10 fold dilution: 20.0ppm / 10 = 2ppm F-

4 fold dilution: 20.0ppm / 4 = 5ppm F-

Area for no dilution: 45.077 x .171 = 7.708167 µS*min

Area for 10 fold dilution: 4.564 x .178 = .812392 µS*min

Area for 4 fold dilution: 11.389 x 0.162 =1.845018 µS*min

NaCl: .0991g NaCl / 2L x 1mol NaCl / 58.44g NaCl x 1 mol Cl- / 1 mol NaCl x 35.453g Cl- x 1000mg / 1g = 30.050ppm Cl-

10 fold dilution: 30.050ppm Cl- / 10 = 3.0050ppm Cl-

4 fold dilution: 30.050ppm Cl- / 4 = 7.5125

Area for no dilution: 39.408 x .205 = 8.07864 µS*min

Area for 10 fold dilution: 3.008 x .214 = .643712 µS*min

Area for 4 fold dilution: 8.103 x .21 = 1.70163 µS*min

NaH2PO4 x 1H2O: .4348g / 2L x 1 / 137.99g x 1 / 1 x 94.9714g x 1000 = 149.63ppm

10 fold dilution: 149.63ppm / 10 = 14.963ppm PO43-

4 fold dilution: 149.63ppm / 4 = 37.4075ppm PO43-

Area for no dilution: 29.592 x .479 = 14.174568 µS*min

Area for 10 fold dilution: 2.267 x .462 = 1.047354 µS*min

Area for 4 fold dilution:6.298 x .455 = 2.86559 µS*min

Na2SO4: .3173g / 2L x 1/142.04g x 1 / 1 x 96.06g x 1000 = 125.6ppm

10 fold dilution: 125.6ppm / 10 = 12.56ppm SO42-

4 fold dilution: 125.6ppm / 4 = 31.4ppm SO42-

Area for no dilution: 40.704 x .632 = 25.724928 µS*min

Area for 10 fold dilution: 3.282 x .598 = 1.962636 µS*min

Area for 4 fold dilution: 9.004 x .591 = 5.321364 µS*min

Unknown Solution #2:

Fluoride: Area = 9.56 x .166 = 1.58696 µS*min

Best fit equation: y= 0.3846x; x = concentration

1.58696 / .3846 = 4.12626105ppm

Chloride: Area: 7.182 x .223 = 1.601586 µS*min

Best fit equation: y = 0.2659x

1.601586 / .2659 = 6.023264385ppm

Phosphate: Area: 4.887 x .457 = 2.233359 µS*min

Best fit equation: y = 0.0934x

2.233359 / .0934 = 23.9117666ppm

Sulfate: Area: 7.149 x .593 = 4.239357 µS*min

Best fit equation: y = 0.2023x

4.239357 / .2023 = 20.95579338ppm

Seawater 1:

Chloride: Area: 22.315 x .202 = 4.50763 µS*min

Original Chloride: 4.50763 / .2659 = 16.95235051ppm / .0009998 = 16955.74166ppm

Sulfate: .686 x .6 = .4116 µS*min

Original Sulfate: .4116 / .2023 = 2.034602076ppm / .0009998 = 2035.009078ppm

Seawater 2:

Chloride: 23.076 x .202 = 4.661352 µS*min

Original Chloride: 4.661352 / .2659 = 17.5304701ppm / .0009998 = 17533.9769ppm

Sulfate: .697 x .420291 = 2.077563025 µS*min

Original Sulfate: 2.077563025 / .2023 = 2.077563025ppm / .0009998 = 2077.978621ppm

Graphs and Chromatograms:

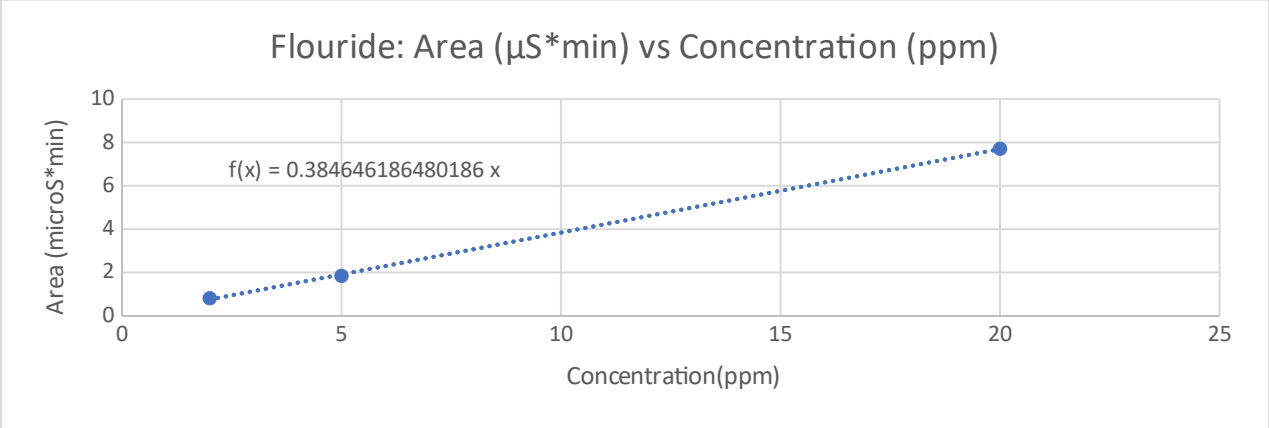

Figure 1: Fluoride ACPS Graph

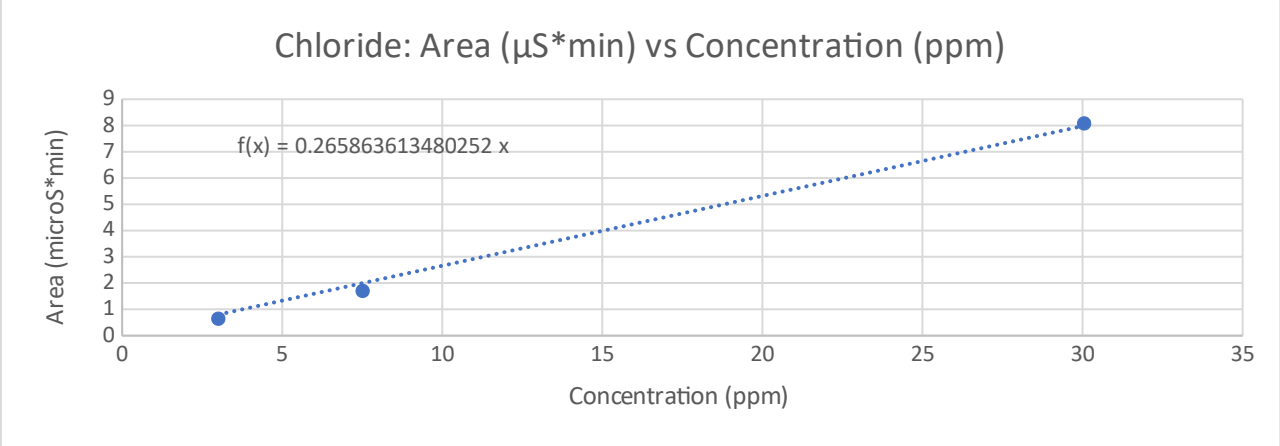

Figure 2: Chloride ACPS Graph

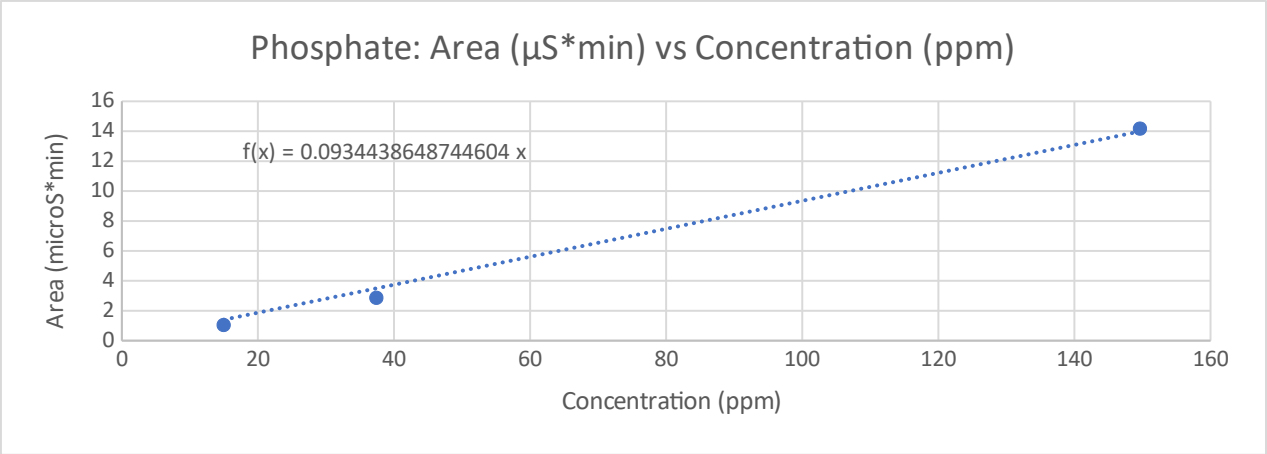

Figure 3: Phosphate ACPS Graph

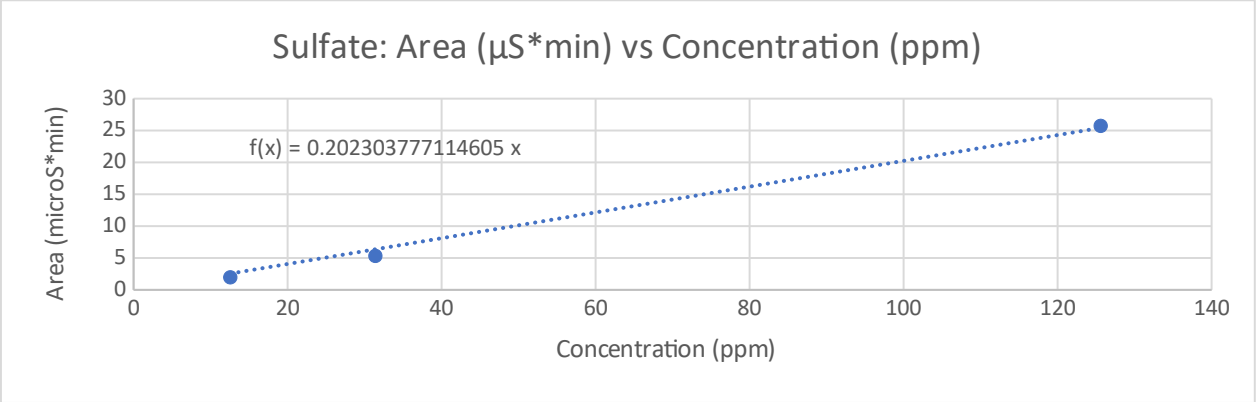

Figure 4: Sulfate ACPS Graph

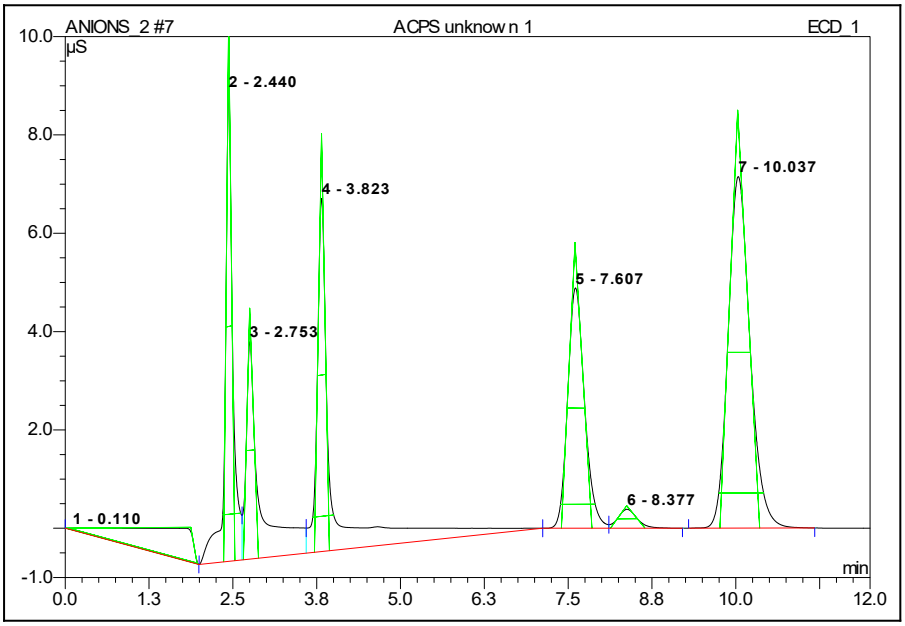

Figure 5: Unknown Chromatogram

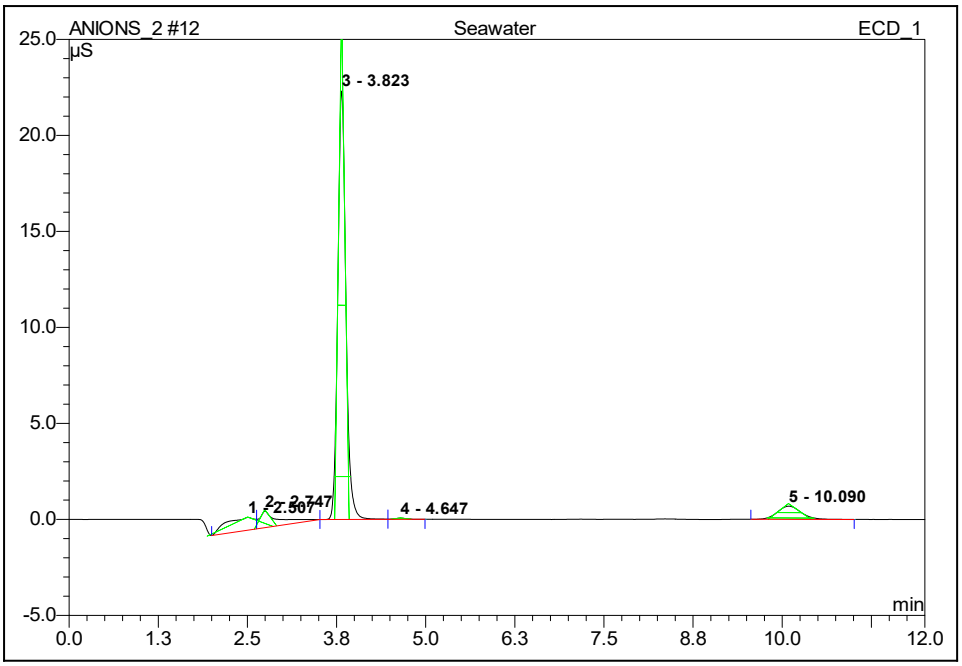

Figure 6: Seawater 1 Chromatogram

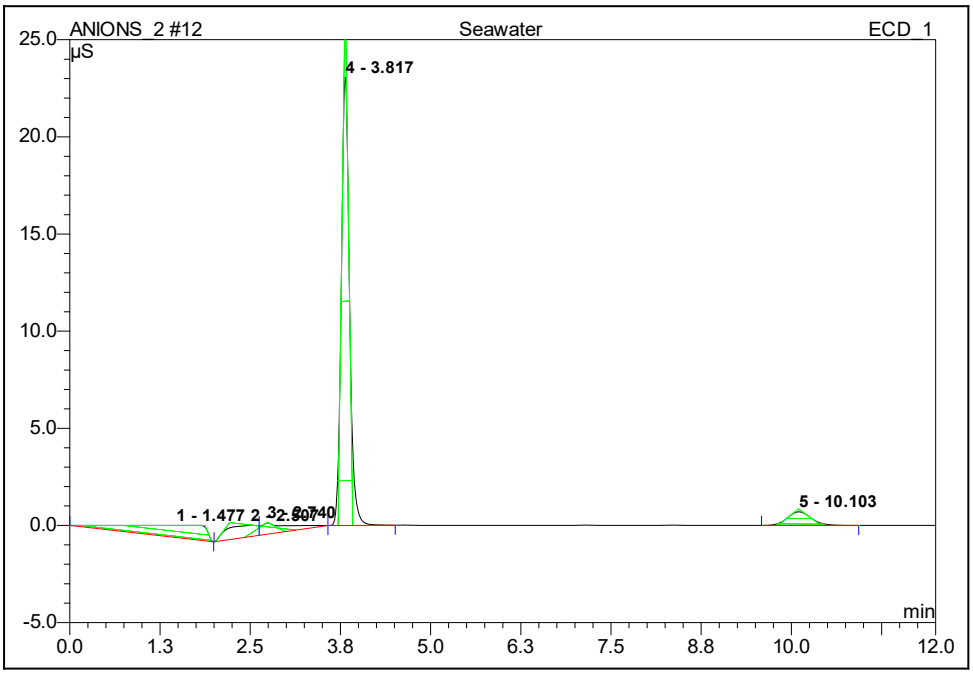

Figure 7: Seawater 2 Chromatogram

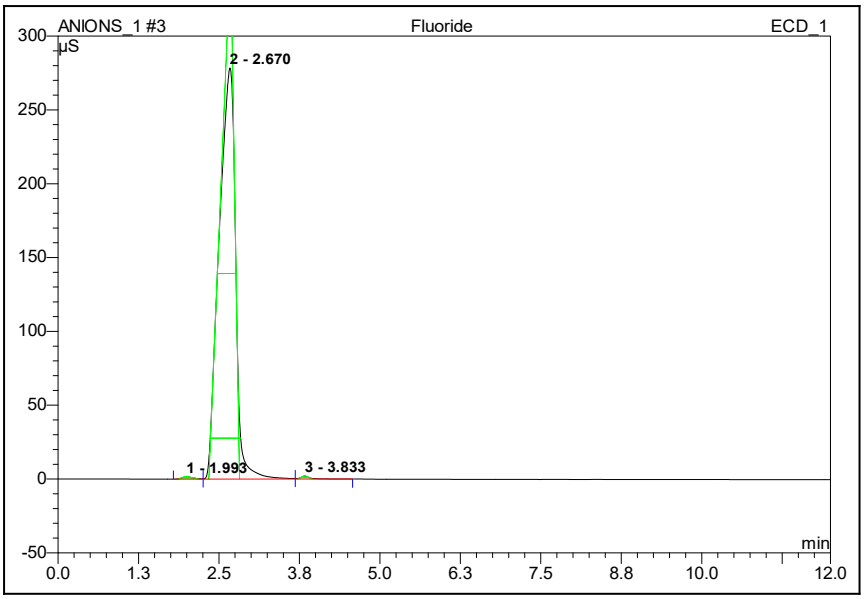

Figure 8: Fluoride Chromatogram

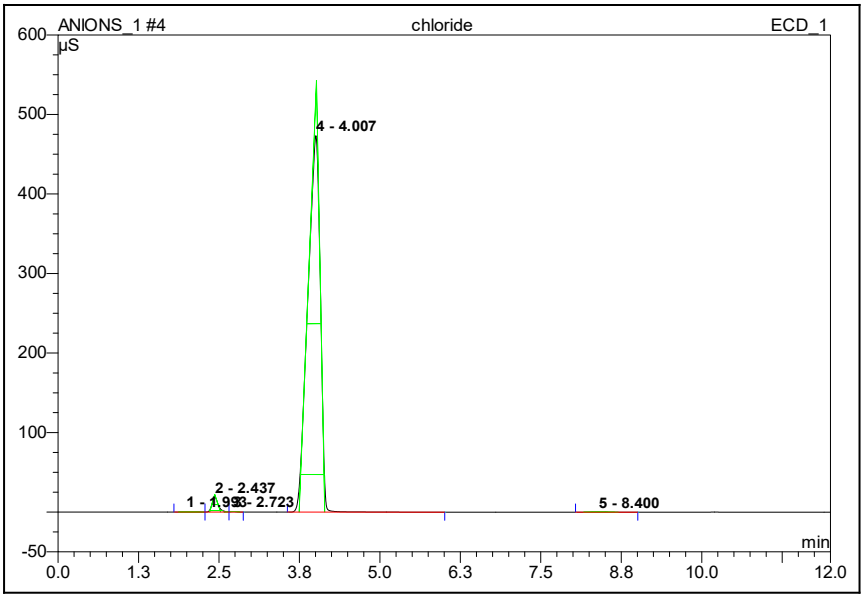

Figure 9: Chloride Chromatogram

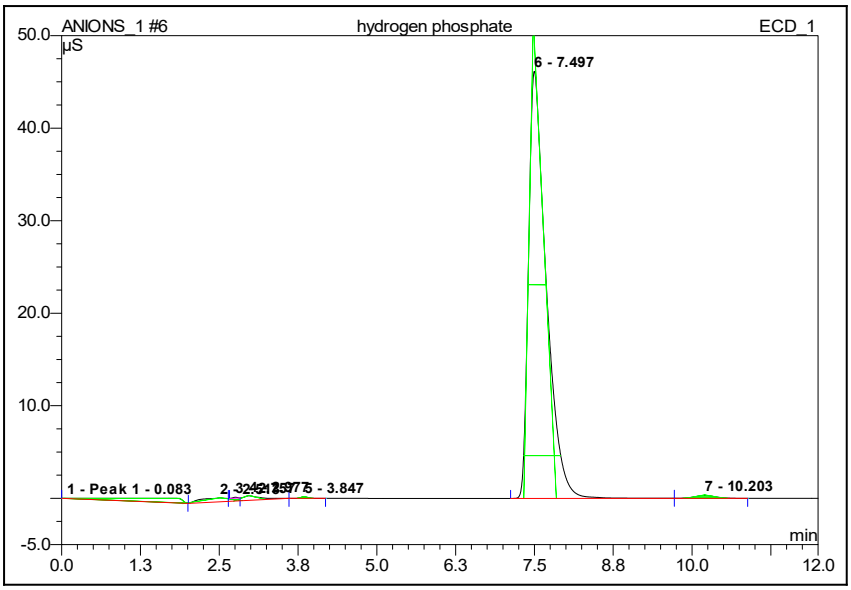

Figure 10: Phosphate Chromatogram

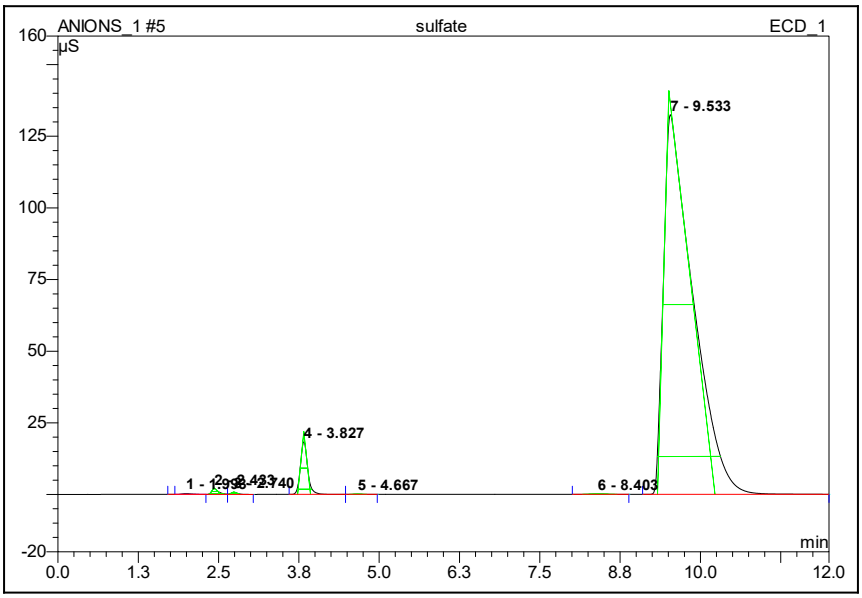

Figure 11: Sulfate Chromatogram

Compared to the gravimetric analysis lab, the average amount of chloride was 18890ppm to 17244.85928ppm, which is about the same result. For the sulfate amount, the average amount of sulfate was 2369.64ppm to 2056.49385ppm, which is also about the same result from the lab. Meanwhile, compared to the LEO-15, Chloride is 18.980ppt and this lab reports it to be 17244.86ppt and sulfate to be 2560ppt and this lab states it to be 2369ppt.

Lab Questions:

1) A blank is used to correct instrumental error.
2) Detector works by measuring conductivity of the solution.

3) The solution that is going to be made is going to be diluted by the deionized water.
4) The factor of dilution is 1000 before pipette calibration.