REPORT FORM FOR EXPERIMENT 6

Name:	Student No.:
Demo Group:	Date of Expt.:
Lab Partners:	

Objective:
– To determine the anion content of a water sample by ion chromatography

Procedure:
– Separation by ion chromatography on an anion exchange column with a $Na_2CO_3/NaHCO_3$ buffer as mobile phase and suppressed conductivity detection at 50 μS (full-scale). Calibration using standard solutions of all ions of interest, followed by direct injection of filtered water samples; all injection volumes 20 μL

Sample:

NB: Elution order is, from first to last: (1) F^- (2) Cl^- (3) Br^- (4) NO_3^- (5) HPO_4^{2-} (6) SO_4^{2-}.

Summary: *Copy your calculated results here after completing the rest of the report form*

Anion	Peak area (μV.s)			$C \pm$ uncertainty (mg/L)
	Run 1	**Run 2**	**Mean**	
F^-				
Cl^-				
Br^-				
NO_3^-				
PO_4^{3-}				
SO_4^{2-}				

Where any other anions present in your sample?

Signature:

Calibration data:

- For *each* anion in the standard mixture, attach a separate properly-labelled graph of peak area (y-axis) vs. concentration (x-axis), showing the best-fit straight line obtained by linear regression analysis. (See the calibration tutorial on the course web site, Lab. notes page.)

- Determine the equation of the best-fit straight line ($y = bx + a$), correlation coefficient (r), and standard deviation of the regression, slope and intercept ($s_{y/x}$, s_b, and s_a, respectively) for each anion. (See the calibration tutorial on the course web site, Lab notes page.)

- Calculate the limit-of-detection (*Clod*) for each anion from the calibration curves and regression data: the l.o.d. is here defined as the concentration that gives rise to a signal equal to the intercept plus three times the standard deviation of the regression, *i.e.* the value of x for $y = a + 3s_{y/x}$.

Fluoride: *summarise the best-fit equations and regression statistics*

Equation:

r:	**s$_{y/x}$**:

Limit-of-detection: *calculate the minimum detectable area & concentration*

$y_{lod} = a + 3s_{y/x} =$

$\therefore C_{lod} =$

Chloride: *summarise the best-fit equations and regression statistics*

Equation:

r:	**s$_{y/x}$**:

Limit-of-detection: *calculate the minimum detectable area & concentration*

$y_{lod} = a + 3s_{y/x} =$

$\therefore C_{lod} =$

Bromide: *summarise the best-fit equations and regression statistics*

Equation:

r:	**s$_{y/x}$**:

Limit-of-detection: *calculate the minimum detectable area & concentration*

$y_{lod} = a + 3s_{y/x} =$

$\therefore C_{lod} =$

Calibration Curves and Regression Analysis (cont.):

Nitrate: *summarise the best-fit equations and regression statistics*	
Equation:	
r:	$s_{y/x}$**:**
Limit-of-detection: *calculate the minimum detectable area & concentration* $y_{lod} = a + 3s_{y/x} =$	
$\therefore C_{lod} =$	
Phosphate: *summarise the best-fit equations and regression statistics*	
Equation:	
r:	$s_{y/x}$**:**
Limit-of-detection: *calculate the minimum detectable area & concentration* $y_{lod} = a + 3s_{y/x} =$	
$\therefore C_{lod} =$	
Sulphate: *summarise the best-fit equations and regression statistics*	
Equation:	
r:	$s_{y/x}$**:**
Limit-of-detection: *calculate the minimum detectable area & concentration* $y_{lod} = a + 3s_{y/x} =$	
$\therefore C_{lod} =$	

Sample Analysis:

Use the regression data to determine the concentrations of all anions identified as being present in your sample. **Note:** if the calculated sample concentration is *less than* the limit-of-detection for that anion, note this clearly on the form. Use the information in the linear regression tutorial from the course web site to calculate the uncertainty in the concentration obtained by interpolating the mean peak area as a 95% confidence interval. If you only have one run for your sample, mark the second column as 'N/A' and enter the standard error in the interpolated value. Remember to account for any sample dilution made to get the peaks on scale in **both** your calculation **and** your error propagation. Attach copies of your calculations to this report form, along with your graphs and discussion. Fill in the following table for **one** of the anions in your sample:

Specimen Calculation:

Analyte:	
Mean peak area:	
Interpolated concentration (mg/L):	
Standard error in this value s_{x0} (mg/L):	
Simple dilution factor (*if applicable*):	
Reported analyte concentration (mg/L):	
Reported uncertainty (mg/L):	

Show your error propagation for the dilution (if applicable):

General Discussion:

Write up your answers to the following questions and attach them, along with your titration curves, to your report form. It is **not** necessary to write an extended answer for each question: you should aim to answer each question in no more than ~50 words.

1. The baseline of your chromatograms shows a negative excursion shortly before the first anion peak (F^-). Why is this?

2. What determines the elution order of the anions in this experiment? Be brief!

3. The separation between the fluoride and chloride peaks could be improved simply by using a longer column that was otherwise identical. What affect would this have on the time required to perform each chromatographic run for the six-component anion standard?

4. The limit of detection could be improved by using a more sensitive setting for the detector (a full-scale reading of 20 μS per 1000 mV instead of 50 μS per 1000 mV). Would this work for the sample you analysed? Why or why not?

5. If you to analyse only a small number of samples for the common halides, would you use ion chromatography or a set of ion-selective electrodes? Briefly explain your reasons.