

## REPORT FORM FOR EXPERIMENT 6

<b>Name:</b>	<b>Student No.:</b>
<b>Demo Group:</b>	<b>Date of Expt.:</b>
<b>Objective:</b> – To determine the anion content of a water sample by ion chromatography	
<b>Procedure:</b> – Separation by ion chromatography on an anion exchange column with a $\text{Na}_2\text{CO}_3/\text{NaHCO}_3$ buffer as mobile phase and suppressed conductivity detection at 50 $\mu\text{S}$ (full-scale). Calibration using standard solutions of all ions of interest injected in triplicate, then direct injection of water samples (injection volume 20 $\mu\text{L}$ )	
<b>Sample:</b>	

**NB** elution order is, from first to last: (1)  $\text{F}^-$  (2)  $\text{Cl}^-$  (3)  $\text{NO}_2^-$  {if present} (4)  $\text{Br}^-$  (5)  $\text{NO}_3^-$  (6)  $\text{HPO}_4^{2-}/\text{PO}_4^{3-}$  (7)  $\text{SO}_4^{2-}$ .

### 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 (l.o.d.) 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  $y = a + 3s_{y/x}$ .

## Calibration Curves and Regression Analysis:

<b>Fluoride:</b> summarise the best-fit equations and regression statistics			
<b>Equation:</b>			
<b>r:</b>	<b>S<sub>y/x</sub>:</b>	<b>S<sub>b</sub>:</b>	<b>S<sub>a</sub>:</b>
<b>Limit-of-detection:</b> calculate the minimum detectable area & concentration $y_{lod} = a + 3s_{y/x} =$			
$\therefore C_{lod} =$			

<b>Chloride:</b> summarise the best-fit equations and regression statistics			
<b>Equation:</b> show calculations			
<b>r:</b>	<b>S<sub>y/x</sub>:</b>	<b>S<sub>b</sub>:</b>	<b>S<sub>a</sub>:</b>
<b>Limit-of-detection:</b> calculate the minimum detectable area & concentration $y_{lod} = a + 3s_{y/x} =$			
$\therefore C_{lod} =$			

<b>Bromide:</b> summarise the best-fit equations and regression statistics			
<b>Equation:</b>			
<b>r:</b>	<b>S<sub>y/x</sub>:</b>	<b>S<sub>b</sub>:</b>	<b>S<sub>a</sub>:</b>
<b>Limit-of-detection:</b> calculate the minimum detectable area & concentration $y_{lod} = a + 3s_{y/x} =$			
$\therefore C_{lod} =$			

### Calibration Curves and Regression Analysis (cont.):

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

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

<b>Sulphate:</b> summarise the best-fit equations and regression statistics			
<b>Equation:</b>			
<b>r:</b>	<b>S<sub>y/x</sub>:</b>	<b>S<sub>b</sub>:</b>	<b>S<sub>a</sub>:</b>
<b>Limit-of-detection:</b> 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, put the value in parentheses (). 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. If you only have one run for your sample, mark the second column as 'N/A' and use the formula for estimating the uncertainty in a single interpolated value.

Anion	Peak area ( $\mu\text{V}\cdot\text{s}$ )			Conc'n (ppm) $\pm$ uncertainty
	Run 1	Run 2	Mean	
F <sup>-</sup>				
Cl <sup>-</sup>				
Br <sup>-</sup>				
NO <sub>3</sub> <sup>-</sup>				
PO <sub>4</sub> <sup>3-</sup>				
SO <sub>4</sub> <sup>2-</sup>				

**Discussion Questions:** *attach your answers on a separate sheet.*

- (1) The baseline of your chromatograms shows a negative excursion shortly before the first anion peak (F<sup>-</sup>). Why is this?
- (2) What determines the elution order of the anions in this experiment? Be brief! (*Maximum of 50 words*)