

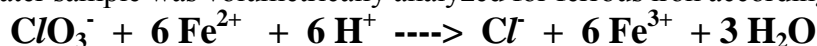
## CHEMISTRY 311 - Mid Term

ANSWER ALL QUESTIONS IN BOOKLETS PROVIDED. SHOW ALL WORK.  
TOTAL MARKS 40.

1. a) What volume of a 50.0  $\mu\text{g/L PO}_4^{3-}$  - P stock solution is required to prepare 250.0 mL of a 1.00  $\mu\text{g/L PO}_4^{3-}$  - P standard solution? [2]

b) A researcher needs to analyze water samples for phosphorous down to levels of 0.050  $\mu\text{M}$ . A supplier of an Ion Chromatograph (IC) reports a detection limit of 5 ppb  $\text{PO}_4^{3-}$ . Does the IC meet the detection limit requirements of the researcher? [2]

2. A wastewater sample was volumetrically analyzed for ferrous iron according to;



a) If a 100. mL wastewater sample required 0.84 mL of 0.100 N  $\text{KClO}_3$  titrant to reach the end point, what is the concentration of  $\text{Fe}^{2+}$  in units of mg/L  $\text{Fe}^{2+}$ ? [2]

b) If the titration was carried out in a class A, 50.0 mL burette (individual reading error  $\pm$  0.02 mL), what is the relative uncertainty in the final answer? [2]

c) What procedural modifications can you suggest to reduce the relative uncertainty in this determination? [2]

d) What mass of potassium chlorate,  $\text{KClO}_3$ , (molar mass 122.5  $\text{g mol}^{-1}$ ) is required to prepare 2.00 L of the titrant solution? [2]

3. A *Standard Reference Material* is certified to contain 94.6 ppm of an organic contaminant in soil. Your analysis gives values of 98.6, 98.4, 97.2, 94.6 and 96.2 ppm. Do your results differ from the expected result at the 95% confidence level? [3]

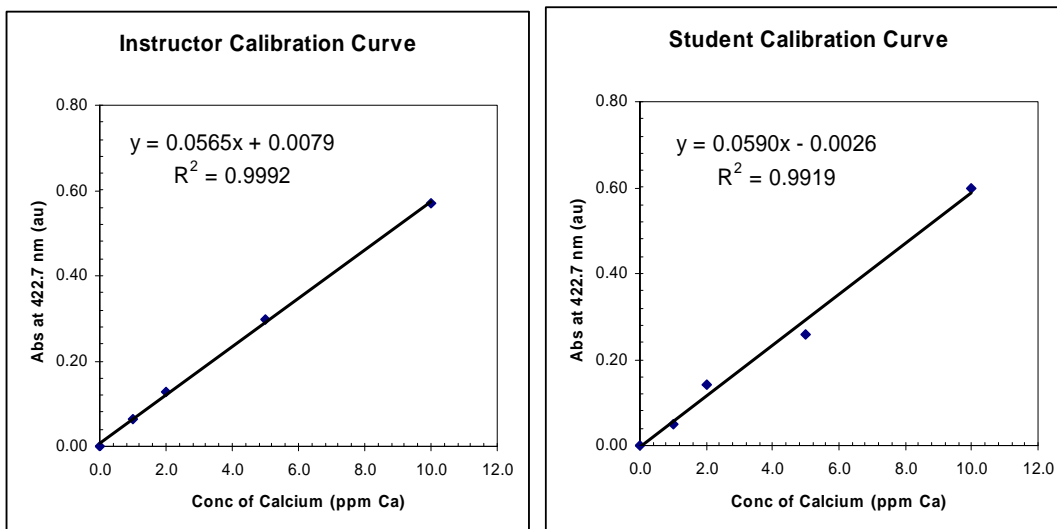
4. During the field analysis of total hardness using a Hach Digital Titrator™ method a 100. mL sample is titrated with a 0.800 M ethylenediaminetetraacetate ( $\text{EDTA}^{4-}$ ), which forms a 1:1 complex with  $\text{Ca}^{2+}$ . A student inadvertently adds one drop ( $\sim 50 \mu\text{L}$ ) of titrant to the sample before carrying out the titration. Estimate the sign and the magnitude of the error introduced in ppm  $\text{CaCO}_3$ . [4]

5. The following procedures may be included in a sampling and analysis programme. Indicate the rationale for THREE of the procedure. [3]

- The sample is filtered (0.45  $\mu\text{m}$ ) and 2 mL/L of  $\text{HNO}_3$  is added
- A *spike* of isotopically labelled analyte is added prior to sample preparation steps
- Every 15<sup>th</sup> lab analysis is of a *certified reference material*
- A deionized water *blank* is collected at the sampling site and transported to the lab for analysis

6. In the determination of Calcium by flame atomic absorption spectroscopy, the following calibration curves were obtained. [3]

- Determine the concentration of Calcium for an unknown sample with an absorbance of 0.440 au.
- Comment on the relative precision and accuracy of results generated using these curves.



7. A blood serum sample was analyzed for potassium by flame atomic emission spectrometry (FAES) using the method of standard additions. In FAES, the signal obtained is directly proportional to analyte concentration. A 0.500 mL aliquot of serum was placed in each of two 5.00 mL volumetric flasks. In one flask, a 20.0  $\mu\text{L}$  spike of 0.0500 M KCl was added. Both flasks were then filled and mixed by inversion. The signal for the unspiked flask was 32.1 a.u. and for the spiked flask 58.6 a.u. respectively. What is the potassium concentration in the original sample? [4]

8. An HPLC analysis of a combined standard of 25.0  $\mu\text{M}$  Valium™ (MW = 284.5 g/mol) and 10.0  $\mu\text{M}$  nitro-diazepam (internal standard) gave signal areas of 4322 a.u. and 1458 a.u., respectively. When a 100. mg hair sample was digested (dissolved), and analyzed for Valium, the digest (15.00 mL) was spiked with 100.0  $\mu\text{L}$  of 20.0 mM nitro-diazepam, and subjected to HPLC analysis. The signal areas obtained for Valium and nitro-diazepam were 152 a.u. and 110 a.u., respectively. Determine the Valium concentration in the digest sample ( $\mu\text{M}$ ) and the original hair sample (mg/g). [5]

9. Briefly describe the difference for any THREE of the following, providing common units of reporting in each case. [6]

- Turbidity versus Total Suspended Solids
- Total Alkalinity versus pH
- Total Dissolved Solids versus Specific Conductivity
- Dissolved Oxygen versus Oxidation Reduction Potential

**CHEM 311**  
**INFORMATION SHEET**

$$s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{N-1}}$$

$$\sigma = \sqrt{\frac{\sum (x_i - \bar{x})^2}{N}}$$

$$CL \text{ for } \mu = \bar{x} \pm \frac{ts}{\sqrt{N}}$$

$$CL \text{ for } \mu = \bar{x} \pm \frac{z\sigma}{\sqrt{N}}$$

$$Q_{\text{exp}} = \frac{\text{gap}}{\text{range}}$$

$$|\bar{x}_1 - \bar{x}_2| = \frac{\pm ts_{\text{pooled}}}{\sqrt{\frac{N_1 N_2}{N_1 + N_2}}}$$

$$|\bar{x}_1 - \mu| = \frac{\pm ts_{\text{pooled}}}{\sqrt{\frac{N_1 N_2}{N_1 + N_2}}}$$

$$s_{\text{pooled}} = \sqrt{\frac{\sum (x_i - \bar{x})^2 + \sum (x_j - \bar{x}_2)^2 + \dots}{N_1 + N_2 + \dots - N_{\text{subsets}}}}$$

**OR**

$$R_m = R_i \left( \frac{W_m}{W_x + W_i} \right)$$

$$s_{\text{pooled}} = \sqrt{\frac{\sum s_1^2 (N_1 - 1) + s_2^2 (N_2 - 1) + \dots}{N_1 + N_2 + \dots - N_{\text{subsets}}}}$$

$$N = 16 \left( \frac{t_R}{W} \right)^2$$

$$K = \frac{k' V_M}{V_S}$$

$$\frac{A_x}{A_{x+s}} = \frac{[X]}{[X] + [S]_f}$$

$$t_{1/2} = \frac{0.693}{\lambda}$$

$$R_x = k w_x$$

$$R_{x+s} = k(w_x + w_s)$$

$$\frac{N_i}{N_o} = \frac{P_i}{P_o} \exp\left(-\frac{E_i}{kT}\right)$$

$$F = \frac{(A_x/C_x)}{(A_s/C_s)}$$

$$\text{Sin}(r) = m\lambda / b$$

$$\sigma_M = \sqrt{M}$$

$$pH = -\log a_{H^+} \cong -\log [H_3O^+]$$

$$\varepsilon_{\text{cell}} = \varepsilon^\circ + \left\{ \frac{2.303RT}{nF} \right\} \log Q$$

$$R = \frac{\lambda}{\Delta\lambda} = nN$$

$$E = h\nu = \frac{hc}{\lambda}$$

Universal Constants:

$$h = 6.626 \times 10^{-34} \text{ Js}$$

$$c = 2.9979 \times 10^8 \text{ m/s}$$

$$k = 1.3806 \times 10^{-23} \text{ J/K}$$

$$F = 96485 \text{ C/mol}$$

$$R = 8.3145 \text{ J/K}\cdot\text{mol}$$



## Selected Statistical Values

Critical Values for Rejection Quotient

# Data Points	Q <sub>crit</sub> at 95% CL
3	0.970
4	0.829
5	0.710
6	0.625
7	0.568

Table of z-values at Various Confidence Levels

Confidence Level	Z
68%	1.00
90%	1.64
95%	1.96
99%	2.58
99.9%	3.29

Table of t values at 95% probability

# Degrees of Freedom	t-value at 95% CL
2	4.30
3	3.18
4	2.78
5	2.57
6	2.45
7	2.36
8	2.31
9	2.26
10	2.23
11	2.20
12	2.18