Name: \_\_\_\_\_

Section: \_\_\_\_\_

Data and Calculations	Trial 1	Trial 2
Mass of empty calorimeter		=g
Mass of calorimeter + water		g
Mass of calorimeter + water + solid (final mas	ss)	g
Mass of solution		g
Mass of solid (solute)		g
Initial temperature		°C
Final temperature		°C
$\Delta T$		°C
$Q_{solution} = (m \ s \ \Delta T)_{solution}$		J
$Q_{rxn} = -Q_{solution}$		J
Calculate the heat of reaction, $\Delta H$ , per gram o	f solid	
$\Delta H_{\text{reaction}} = \frac{Q_{\text{rxn}}}{\text{mass}_{\text{solute}}}$	/ g (trial 1)* and	J / g (trial 2)*
* <u>Note</u> : These values should be within 5% of e	each other or another tria	al should be done.
Average ΔH per gram J / g		
Molecular Formula of Solid	Molecular We	ight
Calculate the molar heat of solution, $\Delta H$ , for t	his solid. Use the averag	ge ∆H value.
	$\Delta H_{reaction}$	kJ / mol
Calculate the % error for the above value. (Literature values: Na <sub>2</sub> CO <sub>3</sub> $\Delta$ H <sub>reaction</sub> = -28.1 k	xJ / mol; NH4NO3 ΔH <sub>read</sub>	<sub>ction</sub> = +25.7 kJ / mol)
Theoretical $\Delta H_{reaction}$	kJ / mol	% error

#### Post-Lab Questions: Heat of Solution

1. A metal sample weighing 63.2 g with a temperature of 100.0  $^{\circ}$ C was placed in a calorimeter containing 41.0 g of water at 24.5  $^{\circ}$ C. The equilibrium temperature of the water and metal was found to be 35.0  $^{\circ}$ C.

A. What was $\Delta T$ for the water? ( $\Delta T = T_{\text{final}} - T_{\text{initial}}$ )	°C

- B. What was  $\Delta T$  for the metal? \_\_\_\_\_°C
- C. Taking the specific heat of water to be 4.184 J / g  $^\circ$ C, calculate the specific heat of the metal using Eq. 5.

J	/	g	°C

- 2. When 5.00 g of KNO<sub>3</sub> were dissolved in 49.00 g H<sub>2</sub>O at 24.00 °C inside a calorimeter, the temperature of the resulting solution fell to 15.60 °C.
  - A. Is this reaction endothermic or exothermic?
  - B. Calculate the heat lost or gained by the solution chemicals (this is  $Q_{soln}$ ) in the calorimeter.

 $Q_{soln} =$ \_\_\_\_\_ Joules

C. What is  $Q_{rxn}$  for the reaction that occurred?

 $Q_{rxn} =$ \_\_\_\_\_ Joules

D. Calculate the heat of this reaction,  $\Delta H$ , in Joules/g KNO<sub>3</sub>.

 $\Delta H_{rxn} =$  \_\_\_\_\_ Joules / g

E. Calculate the molar heat of this reaction,  $\Delta H$ , in kJ/mol KNO<sub>3</sub>.

 $\Delta H_{rxn} = \___kJ / mole$ 

Data for Part A: Clearly show calculations of	n another space,	(s) = 3.70	J/g°C
Mass of Mg			
Mass of Empty Calorimeter + Lid		= _	
Mass of Calorimeter + Lid + Final Solution _			
Initial Temperature of HCl Solution			
Final Temperature of Solution			
Mass of Final Solution			
$\Delta T$ of Solution			
$\Delta H$ for reaction <b>Part A</b> , kJ/mole Mg			
Average $\Delta H_A$			kJ / mol Mg
Data for Part B: Clearly show calculations or	n another space,	(s) = 3.70	J / g °C
Mass of Empty Calorimeter + Lid		_ = _	
Mass of Calorimeter + Lid + Final Solution _			
Mass of Weighing Dish			
Mass of Weighing Dish + MgO			
Initial Temperature of HCl			
Final Temperature of Solution			
$\Delta T$ of Solution			
Mass of MgO			
Mass of Final Solution			
$\Delta H$ for reaction <b>Part B</b> , kJ / mole MgO			
Average $\Delta H_B$			kJ / mol MgO

#### **Data Analysis and Calculations**

Use Hess's Law and the following information to determine the heat of formation of MgO:

A.	$\begin{array}{l} Mg(s)+2 \; HCl \; (aq) \rightarrow H_2(g) + MgCl_2(aq) \\ (\textbf{Part A}) \end{array}$	$\Delta H_A =$
B.	$\begin{array}{l} MgO(s)+2 \ HCl \ (aq) \rightarrow H_2O(l) + MgCl_2(aq) \\ (\textbf{Part B}) \end{array}$	$\Delta H_B = \_$
C.	Write the formation equation for liquid water	$\Delta H_{f}^{\circ} =$

- D. Write the formation equation for MgO(s)
- E. Use Hess's Law and the above information to calculate the heat of formation for MgO(s). Show all your work.

F. Look up the literature value of  $\Delta H_f^{\circ}$  for MgO and calculate the % error.

Theoretical  $\Delta H_f^{\circ}$  MgO \_\_\_\_\_ kJ / mol % error \_\_\_\_\_

Use appendix for value

- 9. Place the bomb in the calorimeter, attach the ignition wires to the top of bomb, cover the calorimeter by aligning the pilot hole and screw, and turn on the stirrer.
- 10. Wait 5 minutes and observe the temperature at 30 second intervals until a constant equilibrium temperature is achieved (4 constant values). Record this temperature.
- 11. Plug in the wires onto the ignition unit and press the button to ignite your sample.
- 12. Continue to observe the temperature until a constant equilibrium temperature is achieved. Record this temperature.
- 13. Carefully remove the cover and place it in the holder.
- 14. Remove the bomb, equalize the pressure, disassemble and completely dry all parts of the bomb.
- 15. If successive trials are to be run, add a new 2000 mL supply of D.I. water to the bucket and repeat steps 3–14.

#### **Data and Calculations**

Volume of sample	Density of sample		
Initial temperature	Final temperature		

- 1. Write a balanced equation for the combustion reaction of one mole of 1-butanol.
- 2. Calculate the mass (in g) of the 1-butanol sample.
- 3. Calculate the heat of reaction per mole of 1-butanol. *Our calorimeter constant (Heat Capacity)* =  $10.3 \text{ kJ} / {}^{\circ}C$

- 4. Does question #3 solve for  $\Delta E$  (internal energy) or  $\Delta H$  (enthalpy)? Circle your choice and briefly explain.
- 5. Calculate the **ideal** work per mole of alcohol for the combustion reaction at 1 atm and 25  $^{\circ}$ C.
- 6. Give the following values in kJ/mol for CH<sub>3</sub>(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>OH at 1 atm and 25°C: Show your work below for full credit.

$\Delta E =$	$\Delta H =$	w =

7. Use literature values of enthalpy of formation to calculate  $\Delta H_{rxn}$  for the balanced combustion reaction in question #1. Use this as the theoretical value.

- 8. Calculate a % error for this experiment using  $\Delta H_{rxn}$  values from problems 6 and 7 above.
- 9. Why is 1 mL of D.I. water added to the bottom of the bomb chamber?

Name: \_\_\_\_\_

Reaction data from mixture D at three temperatures has been provided for you to determine the energy of activation for the reaction using Equation 2.

# **Data and Calculations**

Mixture	Volume (mL) of:			Time of reaction (sec)			Temp	
	4.0 M acetone	1.0 M HCl	0.0050 M I2	H <sub>2</sub> O	1 <sup>st</sup> run	2 <sup>nd</sup> run	Ave time	°C
А	1.00	1.00	1.00	2.00				
В	2.00	1.00	1.00	1.00				
С	1.00	2.00	1.00	1.00				
D	1.00	1.00	0.50	2.50				
Your Mixture <sup>*</sup>								

Table 1. Reactant Volumes and Reaction Times

\*Note: Must add up to 5.0 mL; water is not required.

Mixture	[acetone] <sub>ini</sub>	$[H^+]_{ini}$	[I <sub>2</sub> ] <sub>ini</sub>	Rate = $[I_2]_{ini}$ / (ave time)
А				
В				
С				
D				
Your				
Mixture*				

#### Part A: Reactant Order Determination

Calculate the value of m (order with respect to acetone) to TWO decimal places. This means you have to use logarithms. Show calculations. Then round off to the nearest integer to get the true value for m.

m =	(2 decimal places)	m =	(nearest integer)
Calculate the value	e of n (order with respect to t	he H <sup>+</sup> ):	
n =	(2 decimal places)	n =	(nearest integer)
Calculate the value	e of p (order with respect to t	he I <sub>2</sub> ):	
p =	(2 decimal places)	p =	(nearest integer)
Write the overall ra	ate law:		

#### Part B: Determination of the Rate Constant, k

Given the values of m, n, and p, calculate the rate constant k (**with correct units**) for each mixture by simply substituting those orders, the initial concentrations, and the observed rate from the Table 2 above into Equation 3.

Table 3. Rate Constant, k

Mixture	А	В	С	D	average	Units of k
k						

Show calculations for k value of Mixture A:

#### Part C: Prediction of Reaction Rate in Your Mixture

Initial concentrations (from Table 2):

 $[acetone]_{ini} \_\_\__ M \qquad [H^+]_{ini} \_\_\__ M \qquad [I_2]_{ini} \_\_\__ M$ 

Calculate the predicted rate based on your determined rate law in Part A as well as your average rate constant (from Part B).

Predicted rate: \_\_\_\_\_ M/sec

Calculate the predicted *time* (in seconds) for reaction based on  $[I_2]_{ini}$  and the value for the predicted rate above using:

Rate =  $[I_2]_{ini}$  / (time)

Predicted time: \_\_\_\_\_\_\_\_ sec

Observed average time for reaction \_\_\_\_\_\_ sec (from Table 1 above)

Calculate the percent difference between the observed and predicted times below:

% difference: \_\_\_\_\_

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# Part D: Energy of Activation Determination PROBLEM SET – Use provided data below

Fill Table 4 below.

#### Table 4. Reaction Rate Data for Mixture **D**.

Temp (°C)	Temp	Time (sec)	Rate = $[I_2]_{ini}$ / (time)
	(Kelvin)		(NOTE: Use $[I_2]_{ini}$ from Mixture D)
10.		208	
22		93	
40.		28	

Fill Table 5 below by calculating the rate constant at each temperature using rate from Table 4 above, initial concentrations for Mixture D (Table 2) and your experimentally determined rate law (from Part A).

Table 5. Calculated Values for Arrhenius Plot

Temperature	k	ln k (y-axis) (two decimal places)	1 / T (x-axis) (Kelvin <sup>-1</sup> )
10 °C			
22 °C			
40 °C			

Construct an Arrhenius plot by graphing  $\ln k \text{ vs. } 1 / \text{T}$ . Find the slope of the best fitting (straight) line through the points. Show your calculations below: Alternatively, you can use MS Excel to construct your graph; write the trendline equation below.

Slope = \_\_\_\_\_ OR Trendline Equation (MS Excel): \_\_\_\_\_

From Equation 2, we see that the slope =  $-E_a / R$  where R = 3.814 J / mol K. Use this relationship to calculate the value of  $E_a$ :

 $E_a = \_\_\_ kJ / mol$ 

## Pre-Lab Questions: The Iodination of Acetone

1. In a reaction involving the iodination of acetone, the following volumes were used to make up the reaction mixture.

 $1.0 \ mL \ 4.0 \ M \ acetone \ + \ 1.0 \ mL \ 1.0 \ M \ HCl \ + \ 1.0 \ mL \ 0.0050 \ M \ I_2 \ + \ 2.0 \ mL \ H_2O$ 

a. Calculate the initial (diluted) concentration of acetone in the reaction mixture.

[acetone]<sub>ini</sub> = \_\_\_\_\_

b. Calculate the initial (diluted) concentration of the hydrogen ion, H<sup>+</sup>, in the reaction mixture.

 $[H^+]_{ini} = \_$ 

c. Calculate the initial (diluted) concentration of iodine, I<sub>2</sub>, in the reaction mixture.

[I<sub>2</sub>]<sub>ini</sub> = \_\_\_\_\_

- 2. Using the reaction mixture in Problem 1, a student found that it took 300 seconds for the color of the  $I_2$  to disappear.
  - a. What was the rate of the reaction?

rate = \_\_\_\_\_

b. Given the rate from Part a and the initial concentrations of acetone,  $H^+$  ion, and  $I_2$  in the reaction mixture, write Equation 3 as it would apply to the mixture.

rate =

c. What are the unknowns that remain in the equation in Part b?

3. A second reaction mixture was made up in the following way:

2.0 mL 4.0 M acetone + 1.0 mL 1.0 M HCl + 1.0 mL 0.0050 M I<sub>2</sub> +  $1.0 \text{ mL } H_2O$ 

a. What were the initial concentrations of acetone,  $H^+$  ion, and  $I_2$  in the reaction mixture?

[acetone]<sub>ini</sub> M;  $[H^+]_{ini}$  M; [I<sub>2</sub>]<sub>ini</sub> M

b. It took 140 seconds for the  $I_2$  color to disappear from the reaction mixture when it occurred at the same temperature as the reaction in Problem 2.

What was the rate of the reaction?

Write Equation 3 as it would apply to the second reaction mixture:

rate =

c. Solve for the value of m, the order of the reaction with respect to acetone. (Use the logarithm method and calculate the value of m to two decimal places and then round to the nearest integer.)

m = (2 decimal places) m = (nearest integer)

Section:

# Table 1. Reaction Rate Data

Reaction Mixture	Time (sec) for color change	Relative Rate = 1/t	$[I^-]_{ini}$	[BrO <sub>3</sub> <sup>-</sup> ] <sub>ini</sub>	$[\mathrm{H}^+]_{\mathrm{ini}}$	Room Temp (°C)
1						
2						
3						
4						
5						

Varying conditions for Mixture 2	Time (sec) for color change	Temp (°C)
Hot: ~ 40 °C		
Cold: ~ 10 °C		
With a catalyst		

The reactant concentrations in the reaction mixture are not those of the stock solutions, since the reagents were diluted by the other solutions. The final volume of the reaction mixture is 25 mL in all cases. We can calculate the concentrations of all reactants in the table above  $(M_1V_1 = M_2V_2)$ .

#### Determination of the Orders of the Reaction

Given the data in the table, the problem is to find the order for each reactant and the rate constant for the reaction. Since we are dealing with relative rates, we can modify Equation 2 to read as follows:

relative rate = k' 
$$[I^{-}]^{m} [BrO_{3}^{-}]^{n} [H^{+}]^{p}$$
 (5)

We need to determine the relative rate constant k' and the orders m, n and p in such a way as to be consistent with the data in Table 1.

The solution to this problem is quite simple once you make a few observations on the reaction mixtures. Each mixture (2 to 4) differs from Reaction Mixture 1 in the concentration of only one species (see table). This means that for any pair of mixtures that includes reaction Mixture 1, there is only one concentration that changes. We can then find the order for the reactant whose concentration was changed.

Write Equation 5 below for Reaction Mixtures 1 and 2, substituting the relative rates and the initial concentrations of  $I^-$ ,  $BrO_3^-$ , and  $H^+$  from Table 1.

Relative rate 1 = \_\_\_\_\_ = k [ ]<sup>m</sup>[ ]<sup>n</sup>[ ]<sup>p</sup> Relative rate 2 = \_\_\_\_\_ = k [ ]<sup>m</sup>[ ]<sup>n</sup>[ ]<sup>p</sup>

Solve for m, the order of the reaction with respect to the  $I^-$ , to two decimal places and then round off to the nearest integer.

m = (2 decimal places) m = (nearest integer)

Apply the same approach to find the value of n, the order of the reaction with respect to the  $BrO_3^-$  ion. Show your set-up.

n = (2 decimal places) n = (nearest integer)

Apply the method once again to find p, the order with respect to the  $H^+$  ion. Show your setup.

p = (2 decimal places) p = (nearest integer)

Having found m, n, and p (nearest integers, the rate law is written as:

rate = \_\_\_\_\_

### Determination of the Value of Relative Rate Constant

The relative rate constant, k' can be calculated by substitution of m, n, p and the known rates and reactant concentrations into Equation 5. Fill Table 2 below by calculating k for Reaction Mixtures 1-4.

Table 2. Reaction Constant, k

Reaction	1	2	3	4	Average	Units of k
k						

Calculations for each k value:

Briefly explain why k should have nearly the same value for each of the Mixtures 1 - 4.

Why?\_\_\_\_\_

Determination of Relative Rate and Reaction Time for Mixture 5

For Reaction Mixture 5, use  $k_{ave}$  from Table 2 above and the appropriate concentrations from Table 1 in Equation 5 to predict (calculate) the relative rate. Then use this value to predict (calculate) the reaction time, t, for Mixture 5. Show your calculations below.

relative rate <sub>predicted</sub>	tpredicted	tobserved
------------------------------------	------------	-----------

Calculate the percent difference of your reaction times.

% difference \_\_\_\_\_

# The Effect of Temperature on Reaction Rate: The Activation Energy

To find the activation energy for the reaction, transfer the reaction times and temperatures onto Table 3 and fill in the remaining boxes using Mixture 2. Since the reactions at the different temperatures all involve the same reactant concentrations, the rate constants, k, for two different mixtures will have the same ratio as the reaction rates themselves for the two mixtures. This means that in the calculation of  $E_a$ , we can use the observed relative rates instead of rate constants.

Table 3. Reaction Rate at Different Temperatures for Mixture 2.

	Room Temp	Hot: ~ 40 °C	Cold: ~ 10 °C
Time (sec)			
for color change			
Actual Temp			
(°C)			
Actual Temp			
(Kelvin)			
1 / Temp			
(Kelvin <sup>-1</sup> )			
Relative Rate = 1/time			
ln (Relative Rate)			

Use the appropriate values from Table 3 and plot  $\ln(\text{relative rate})$  vs 1 / T in Microsoft Excel.<sup>®</sup> Insert a linear trendline and display both the equation and  $r^2$  value of the trendline on the graph. Copy these in the space below. Make certain to include your Arrhenius plot with this report.

Trendline Equation: \_\_\_\_\_

r<sup>2</sup> =\_\_\_\_\_

The slope of the line equals  $-E_a / R$ , where R = 8.314 Joules / (mole K). Use this relationship to calculate the activation energy,  $E_a$ , for your reaction in kJ/mol.

 $E_a = \_\_\_ kJ / mol$ 

The Effect of a Catalyst on Reaction Rate

Room Temperature
Mixture 2

Catalyzed Mixture 2

Time for color to appear (sec)

Would you expect the activation energy,  $E_a$ , for the catalyzed reaction to be greater than, less than, or equal to the activation energy for the uncatalyzed reaction? Briefly explain.

Name:
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### Pre-Lab Ouestions: Iodine Clock Reaction

- 1. A student studied the clock reaction described in this experiment. She set up a reaction mixture by mixing 10.0 mL of 0.010 M KI, 10.0 mL of 0.0010 M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, 20.0 mL of 0.040 M KBrO<sub>3</sub> and 10.0 mL of 0.10 M HCl using the procedure given. It took 40.0 seconds for the color to turn blue.
  - a. She found the concentration of each reactant in the reacting mixture by realizing that the number of moles of each reactant did not change when that reactant was mixed with the others, but that its concentration did. The volume of the mixture was 50.0 mL. Find the initial concentration of each reactant.

 $[I^{-}]_{ini} = \_____ M; \quad [BrO_3^{-}]_{ini} = \____ M; \quad [H^{+}]_{ini} = \____ M$ 

- b. What is the relative rate of the reaction (1/time)?
- 2. The student repeated the experiment using reaction mixture 1 by mixing 10 mL of 0.010 M KI, 10 mL of 0.0010 M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, 10 mL of 0.040 M KBrO<sub>3</sub>, 10 mL of 0.10 M HCl and 10 mL of H<sub>2</sub>O. It took 75 seconds to turn blue.

Find the initial concentration of each reactant after mixing:

 $[I^{-}]_{ini} = \____ M; \qquad [BrO_3^{-}]_{ini} = \___ M; \qquad [H^{+}]_{ini} = \___ M$ 

What is the relative rate of the reaction (1 / time)?

3. Use the information above to determine the order with respect to  $BrO_3^{-}$ .

n = (2 decimal places) n = (nearest integer)

N	ame:
τ,	unit.

## **Data and Calculations**

Wavelength of maximum absorbance is \_\_\_\_\_\_.

$[Co^{+2}]$	Volume of 0.100 M Co(NO <sub>3</sub> ) <sub>2</sub> Stock Solution	<u>ABSORBANCE</u>
<u>0.1000 M</u>	mL	
<u>0.0800 M</u>	mL	
<u>0.0600 M</u>	mL	
<u>0.0400 M</u>	mL	
<u>0.0200 M</u>	mL	
<u>0.0000 M</u>	mL	

UNKNOWN # \_\_\_\_\_ ABSORBANCE \_\_\_\_\_

Path length of the cuvette: \_\_\_\_\_

Using Microsoft Excel, plot a graph of absorbance (y) verses concentration (x). Using the graph plotted from your data and the path length of the cuvette, calculate the extinction coefficient.

Extinction Coefficient:

Solve for the concentration on your unknown solution...

(a) [Co<sup>+2</sup>] \_\_\_\_\_(read from graph)

(b) [Co<sup>+2</sup>] \_\_\_\_\_ (calculate from line equation and slope value)

SHOW CALCULATIONS:

# Post-Lab Questions: Colorimetric of Co<sup>+2</sup>

1. Calculate the transmittance of a solution if its absorbance is 0.352.

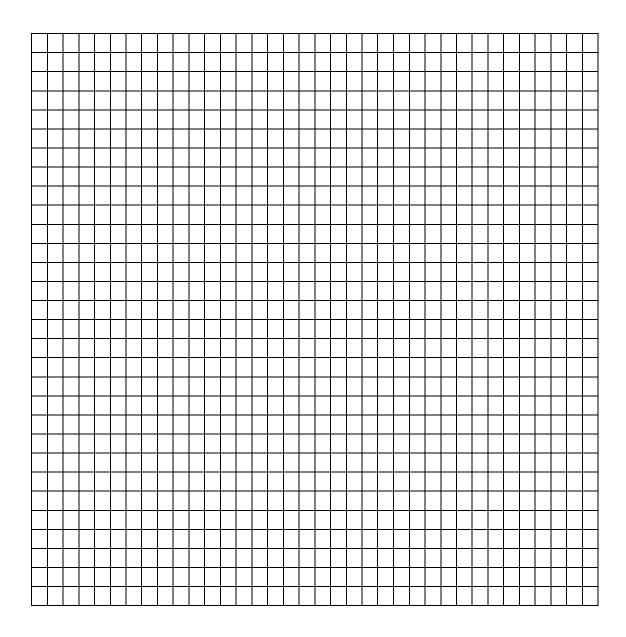
2. Calculate the absorbance of a solution if the transmittance is 0.647.

3. The following absorbance values for four solutions with known MnO<sub>4</sub><sup>-</sup> concentrations were measured using a spectrophotometer:

Solution	[MnO4 <sup>-</sup> ]	Absorbance
1	$0.700 \ge 10^{-4} M$	0.175
2	1.00 x 10 <sup>-4</sup> M	0.250
3	2.00 x 10 <sup>-4</sup> M	0.500
4	3.50 x 10 <sup>-4</sup> M	0.875

- A. Using Microsoft Excel, plot a graph of Absorbance vs. Concentration of  $MnO_4^-$ . Write the trendline linear equation from the plotted graph.
- B. Determine the slope of the graph and include its units.
- C. Determine the concentration of an unknown  $MnO_4^-$  sample whose absorbance is 0.780.

D. Using the graph paper, below, construct a graph of Absorbance vs Concentration of  $MnO_4^-$ . Draw a linear trendline and determine the equation of the line that you drew. How does this compare to the graph that you made using Excel?



**Pre-Lab Questions**: Equilibrium Constant Determination for FeSCN<sup>+2</sup>

- 1. A student mixes 5.00 mL of  $2.00 \times 10^{-3}$  M Fe(NO<sub>3</sub>)<sub>3</sub> with 5.00 mL of  $2.00 \times 10^{-3}$  M KSCN. She finds that in the equilibrium mixture, the concentration of FeSCN<sup>2+</sup> is  $1.40 \times 10^{-4}$  M. Find K<sub>c</sub> for Fe<sup>3+</sup>(aq) + SCN<sup>-</sup>(aq) = FeSCN<sup>2+</sup>(aq).
  - Step 1. Calculate the initial, diluted concentrations of the  $Fe^{3+}$  and  $SCN^{-}$  ions in the total of 10.00 ml solution using  $M_1V_1 = M_2V_2$

[Fe<sup>3+</sup>]\_\_\_\_\_[SCN<sup>-</sup>]\_\_\_\_\_

.

Step 2. Use the initial concentrations of the  $Fe^{3+}$  and  $SCN^{-}$  ions along with the equilibrium concentration of the  $FeSCN^{2+}$  ion and the reaction stoichiometry to determine the equilibrium concentrations of  $Fe^{3+}$  and  $SCN^{-}$ .

	[Fe <sup>3+</sup> ]	+	[SCN <sup>-</sup> ]	$\leftarrow$	[FeSCN <sup>2+</sup> ]
initial					
Δ					
equil					

Step 3. Solve for the value of K<sub>c</sub> for the reaction. (Use Eq. 2 and the results of Step 2.)

K<sub>c</sub> = \_\_\_\_\_

Vol. of 2.00 × 10 <sup>-3</sup> M Vol. of 2.00 × 10 <sup>-3</sup> M Vol. of 1.00 (in mL)         Vol. of 1.01         Vol. of 1.01 <th></th> <th></th>		
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	M Vol. of H <sub>2</sub> O (in mL) Absorbance	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	4.00	
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	3.00	
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	2.00	
5.005.00 $0.00$ Initial ConcentrationsEquilibrium ConcentrationsAFTER MIXING [Fe <sup>+3</sup> ][SCN <sup>-</sup> ][Fe <sup>+3</sup> ][SCN <sup>-</sup> ][Fe <sup>+3</sup> ][SCN <sup>-</sup> ][Fe <sup>+3</sup> ][SCN <sup>-</sup> ]	1.00	
Initial Concentrations     Equilibrium Concentrations       AFTER MIXING     [Fe <sup>+3</sup> ]       [Fe <sup>+3</sup> ]     [SCN <sup>-</sup> ]       [Fe <sup>+3</sup> ]     [SCN <sup>-</sup> ]       [Fe <sup>+3</sup> ]     [SCN <sup>-</sup> ]	0.00	
	n Concentrations + [SCN <sup>-</sup> ] $\Leftrightarrow$ [FeSCN <sup>+2</sup> ]	Kc
<ul> <li>6 ω 4</li> </ul>		
<ul> <li>6 4</li> </ul>		
4		
5		

# Post-Lab Questions: Determination of the Equilibrium Constant for the Formation of $\rm FeSCN^{+2}$

1. Are the  $K_c$  values on the previous page consistent? If not, suggest a reason for any large differences.

2. In carrying out this analysis, we made the assumption that the reactants were reacting as a 1:1 mole ratio, as given by Equation 1. There is no inherent reason why the reaction might not have been a 1:2 mole ratio:

$$\operatorname{Fe}^{3+}(aq) + 2\operatorname{SCN}^{-}(aq) \rightleftharpoons \operatorname{Fe}(\operatorname{SCN})_{2}^{+}(aq)$$
(3)

a. Fill in the equilibrium values in the chart below using your experimental data and this new reaction ratio:

Reaction	$Fe^{+2}(aq) +$	$2 \operatorname{SCN}(aq) \rightleftharpoons$	$Fe(SCN)_2^+(aq)$
test tube 1 mixture			
at equilibrium			
test tube 5 mixture			
at equilibrium			

b. Calculate the value of  $K_c$  using the data from the test tube 1 mixture, assuming that the reaction is actually the one shown in equation 3.

c. Calculate the value of  $K_c$  using the data from the test tube 5 mixture, assuming that the reaction is actually the one shown in equation 3.

d. Compare the  $K_c$  values that you calculated in parts a and b above. Are they consistent? Do you think Reaction 3 is occurring?

# **Pre-Lab Questions**

- 1. A student withdraws 3.00 mL of supernate from a saturated solution of  $KHC_8H_4O_4$  at room temperature (~ 22 °C). This sample was titrated to the phenolphthalein endpoint and 12.85 mL of 0.0997 M NaOH was required.
  - a. Calculate moles of  $HC_8H_4O_4^-$  in the 3.00 mL aliquot.
  - b. Calculate the  $[HC_8H_4O_4^-]_{equil}$  (a.k.a. solubility).

c. What is the value for  $[K^+]_{equil}$ ?

d. Calculate  $K_{sp}$  at 22 °C.

- A similar titration as in #1 above was done for a saturated solution of KHP in 0.500 M KCl also at room temperature. The withdrawn supernate was 5.00 mL (instead of 3.00 mL). The volume of titrant (0.0997 M NaOH) used was 8.35 mL.
  - a. Calculate the new solubility in 0.500 M KCl.

b. Calculate the % decrease in solubility by using equation 5.

3. Two more titrations like the one in #1 above were done at two other temperatures and a plot of ln K<sub>sp</sub> vs 1/T (in Kelvin) was constructed and yielded the trendline equation below. Calculate  $\Delta H^{\circ}_{soln}$  (kJ/mol) and  $\Delta S^{\circ}_{soln}$  (kJ/K·mol).

y = -4480 x + 13.8

 $\Delta H^{\circ}_{soln} = \underline{\qquad } \mathbf{kJ/mol} \quad \Delta S^{\circ}_{soln} = \underline{\qquad } \mathbf{kJ/K'mol}$ 

N	ame:

## **Data and Calculations**

Concentration of standard NaOH: \_\_\_\_\_ M

Sample of sat'd KHP	Temperature (Kelvin)	Vol KHP acid (mL)	Vol NaOH base (mL)	[HC <sub>8</sub> H <sub>4</sub> O <sub>4</sub> <sup>-</sup> ] <sub>eq</sub> (solubility) (M)	[K <sup>+</sup> ] <sub>eq</sub> (M)	K <sub>sp</sub>
At room temp						
warmer						
colder						
At room temp with 0.50M KCl						

Show sample calculations for [HC<sub>8</sub>H<sub>4</sub>O<sub>4</sub><sup>-</sup>]<sub>eq</sub>, [K<sup>+</sup>]<sub>eq</sub> and K<sub>sp</sub> below:

Prepare an Excel<sup>®</sup> chart filling in  $K_{sp}$  and T values. Have the computer program calculate  $ln(K_{sp})$  and 1 / T for the 4 sets of data.

Plot a scatter graph with  $y = ln(K_{sp})$  values and x = 1/T (Kelvin) and display the linear trendline equation of your plot. Attach your graph.

Write out the linear trendline equation \_\_\_\_\_

#### **Post-Lab Questions:**

1. Based on your results at room temperature for saturated KHP in water, calculate the solubility of KHP in g / L.

2. What will happen to the solubility of saturated KHP at room temperature if NaOH is added to the solution? (increase, decrease or no change)? Explain your reasoning.

Additionally, how would adding NaOH affect the K<sub>sp</sub> of KHP at room temperature?

3. Calculate the % decrease in solubility at room temperature between saturated KHP and saturated KHP in 0.50M KCl according to equation 5.

4. Calculate the % difference in K<sub>sp</sub> at room temperature, equation 6. Based on a 10% acceptable difference, are your K<sub>sp</sub> values in water and in KCl the same? If they are not within 10% of each other, propose a specific error that might have caused this significant difference in K<sub>sp</sub>.

5. Plot ln K<sub>sp</sub> vs 1 / T (Kelvin) and determine the linear trendline equation of your plot. Attach your graph.

Linear Trendline Equation:

6. Calculate the  $\Delta H^{\circ}_{soln}$  and  $\Delta S_{soln}^{\circ}$  for the dissolution of solid KHP from the trendline equation of your scatter graph;  $ln(K_{sp})$  vs 1 / T(Kelvin). R is the gas constant 8.314 J  $mol^{-1} K^{-1}$ 

 $\Delta H^{\circ}_{soln} =$ \_\_\_\_\_\_ **kJ / mol**  $\Delta S^{\circ}_{soln} =$ \_\_\_\_\_\_ **kJ / K'mol** 

**Pre-Lab Questions**: Buffers and Determination of Equivalent Mass and  $K_a$  of an Unknown Acid

- 1. What is the equivalent mass of each of the following acids? 1.  $HC_2H_3O_2$ 
  - 2. KHCO<sub>3</sub>
  - $3. \hspace{0.1in} H_2SO_3$
  - 4. H<sub>3</sub>PO<sub>4</sub>
- 2. It is found that 24.6 mL of 0.116 M NaOH is needed to titrate 0.293 g of an unknown acid to the phenolphthalein end point. Calculate the equivalent mass of the acid.

# **Data and Questions**

# Part 1A: pH of Unknown and Buffer Solutions

Enter in the appropriate space the name of the indicator used, the observed color of unknown after addition of the indicator, and the estimated pH value from the pH paper for the unknown.

Liquid Unknown #: \_\_\_\_\_

pH paper estimate: \_\_\_\_\_\_ (3-pH unit range)

Indicator Used	Color of Unknown	Color of Buffer pH =	Color of Buffer pH =	

Estimate pH based on matching of colors = \_\_\_\_\_ (within 0.5 pH unit)

# Part 1B: pH of Acetic Acid Solutions

(<u>Note</u>:  $HAc = HC_2H_3O_2$ , acetic acid).

	1.0 M HC <sub>2</sub> H <sub>3</sub> O <sub>2</sub>	0.10 M HC <sub>2</sub> H <sub>3</sub> O <sub>2</sub>	0.010 M HC <sub>2</sub> H <sub>3</sub> O <sub>2</sub>
рН			
Ka			
% dissociation			

SHOW YOUR CALCULATIONS ON THE NEXT PAGE.

1.0 M HC<sub>2</sub>H<sub>3</sub>O<sub>2</sub> (aq):

0.10 M HC<sub>2</sub>H<sub>3</sub>O<sub>2</sub> (aq):

0.010 M HC<sub>2</sub>H<sub>3</sub>O<sub>2</sub> (aq):

N	ame:

## Part 2: pH of Salt Solutions

1. PREDICT whether each of the salt solutions below is expected to be acidic, neutral, or basic:

	NaCl_		NaC <sub>2</sub> H <sub>3</sub> O <sub>2</sub>	Na <sub>2</sub> CO <sub>3</sub>
	NH4Cl		KNO3	ZnCl <sub>2</sub>
2.	Using	the pH meter imme	ersed in each salt solution	on, determine the actual pH:
	NaCl_		NaC <sub>2</sub> H <sub>3</sub> O <sub>2</sub>	Na <sub>2</sub> CO <sub>3</sub>
	NH <sub>4</sub> Cl	·	KNO <sub>3</sub>	ZnCl <sub>2</sub>
3.	hydrol		ach salt solution. From	NET-IONIC equations for the the net-ionic equation, verify that
	A.	NaCl(aq):		
acidic		Molecular:		
neutra or basic?		Ionic:		
		Net-Ionic:		
	B.	NaC <sub>2</sub> H <sub>3</sub> O <sub>2</sub> (aq)		
		Molecular:		
		Ionic:		
		Net-Ionic:		

C.	Na <sub>2</sub> CO <sub>3</sub> (aq)
	Molecular:
	Ionic:
	<u>Net-Ionic</u> :
D.	NH <sub>4</sub> Cl(aq)
	Molecular:
	Ionic:
	Net-Ionic:
E.	KNO <sub>3</sub> (aq)
	Molecular:
	Ionic:
	Net-Ionic:
F.	ZnCl <sub>2</sub> (aq)
	Molecular:
	Ionic:
	Net-Ionic:

## Part 3: Determination of K<sub>a</sub> and Properties of a Buffer

Solid Unknown Number: \_\_\_\_\_

- 1. Original pH of the half neutralized solution:
- 2. Calculate K<sub>a</sub> of the Weak acid:
- 3. Fill in table:

	tap water (original pH)	tap water (pH after)	Buffer (original pH)	Buffer (pH after)
addition of				
0.1 M HCl				
addition of				
0.1 M NaOH				

4. How does the table above show that the half-neutralized solution is indeed a buffer?

- 5. Using the data on your table above, comment on the buffering ability of your halfneutralized solution in comparison to the tap water.
- 6. Comment on the comparison between adding a strong acid vs a strong base to your buffer solution (i.e. is this solution more resistant to an increase or a decrease in pH?).

## Part 4: Determination of the Equivalent Mass of an Unknown Acid

Given: \_\_\_\_\_ M NaOH

#### Fill in the table below

Sample	Mass unknown acid (g)	Volume NaOH used (mL)	Volume NaOH used (L)	Mol NaOH equal to Mol H <sup>+</sup>	Gram Equivalent Mass of Acid (g/mol H <sup>+</sup> )
Trial 1					
Trial 2					
					Average GEM: g/mol H <sup>+</sup>

Show sample calculations below

# Part 5: Determination of the K<sub>a</sub> and Equivalent Mass of an Unknown Acid using LabQuest Mini

Use the same unknown sample as part 4.

Solid Unknown Number: \_\_\_\_\_

- Determine the approximate mass desired to reach the equivalence point in approximately 15 ml of NaOH added.
   Approximate mass to use = (mass of acid/volume of base)<sub>part 4</sub> x 15ml desired
- 2. Mass accurately weighed into a clean, dry 150 ml beaker.
- 3. Using the graph, determine the volume and pH of titrant at equivalence point.

Volume \_\_\_\_\_ pH \_\_\_\_\_

4. Using the graph, determine the volume and pH at the half-equivalence point.

Volume \_\_\_\_\_ pH \_\_\_\_

5. Solve for the pK<sub>a</sub>, K<sub>a</sub>, and gram equivalent mass of your unknown acid using the data collected in part 5.

Unknown #: \_\_\_\_\_

6. Calculate the average of all three GEM that you determined (two from part 4 and one from part 5).

7. Why is the equivalence point NOT at pH 7?

- 8. Identify the following areas on the weak acid/strong base titration curve.
  - A) Weak acid
  - B) Buffer zone
  - C) Equivalence point, salt
  - D) Strong base zone
  - E) Half equivalence point

**Post-Lab Questions**: Buffers and Determination of Equivalent Mass and  $K_a$  of an Unknown Acid

1. A buffer was prepared by mixing 50.0 mL of 0.10 M HX and 25.0 mL of 0.10 M NaOH. The  $K_a$  of HX is 1.5 x 10<sup>-6</sup>. Calculate the pH of this buffer.

2. The following values were experimentally determined for the titration of 0.145 g of a weak acid with 0.100 M NaOH:

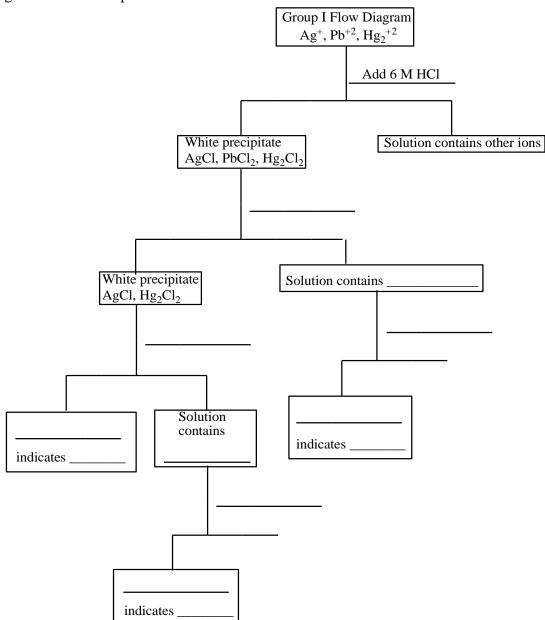
Volume of NaOH, mL	pН
0.0	2.88
5.0	4.15
10.0	4.58
12.5	4.76
15.0	4.93
20.0	5.36
24.0	6.14
24.9	7.15
25.0	8.73
26.0	11.29
30.0	11.96

- A. Construct a titration curve (pH vs Volume of NaOH).
- B. Examine the graph for the required volume to reach the equivalence point?
- C. Examine the graph and state the pH at the half-equivalence point?
- D. Determine the  $K_a$  of the acid.
- E. Calculate the gram equivalent mass of the acid.

3. The following acid-base indicators are available to indicate the end point of this weak acid/strong base titration. Which of them would be most appropriate? Explain.

<b>Indicator</b>	Color Change		pH Transition
	Acid Form	<b>Base Form</b>	
Bromphenol blue	yellow	blue	3.0-5.0
Bromthymol	blue	blue	6.0-7.6
Thymol blue	yellow	blue	8.0-9.6

It is possible to summarize the directions for analysis of the Group I cations in what is called a flow diagram. In the diagram, vertical lines link successive steps in the procedure. Reactant cations or reactant substances containing the ions are at the top end of each line and products formed are at the bottom end. On the product end, a horizontal line separates the <u>solid products on the left</u> and the <u>solution products on the right</u>. Reagents and conditions used to carry out each step are placed alongside the lines. A partially completed flow diagram for the Group I ions follows:



Use this diagram as a brief guide to the procedure. Complete the flow diagram above by directly recording your observations on your known (in the boxes) and unknown (beside the boxes), perhaps using different colored markers.

#### **Experiment Results:**

UNKNOWN NUMBER \_\_\_\_\_ IONS PRESENT \_\_\_\_\_

#### Post-Lab Questions: Group I Cations

1. A solution may contain Ag<sup>+</sup>, Pb<sup>2+</sup>, and Hg<sub>2</sub><sup>2+</sup>. A white precipitate forms on addition of 6 M HCl. The precipitate is partially soluble in hot water. The solid remaining after treatment with hot water turns black on addition of 6 M NH<sub>3</sub>. Which of the ions are present, which are absent, and which remain undetermined? State your reasoning below. <u>NOTE</u>: simply listing ions below without the appropriate reasoning will NOT earn you any credit!

Present

Absent \_\_\_\_\_

In Doubt \_\_\_\_\_

**Pre-Lab Assignment:** Prepare a complete flow diagram for the separation and identification of the various cations presented in this experiment.

#### **Experiment Results:**

UNKNOWN\_\_\_\_\_ IONS PRESENT\_\_\_\_\_

### **Post-Lab Questions:**

1. The precipitates of iron(III) ion and manganese(II) ion are hydroxides. What is the purpose of adding 6 M HNO<sub>3</sub>(aq) in step 3 of the Group A analysis?

2. In step 2 of Group B, Na<sub>2</sub>SO<sub>4</sub> is added. A white precipitate indicates the presence of barium ion. Look up and write down the appropriate K<sub>sp</sub> values and suggest a reason why a calcium precipitate does not form along with the barium precipitate.

3. Describe any modifications (if any) that you discovered that worked for your analysis. How would you improve this experiment?

Section:

4. Test for the Iodide Ion

Acidify a 2 mL sample of a new test solution by adding 6 M HCl. Add 1 mL of 0.1 M FeCl<sub>3</sub> to oxidize any I<sup>-</sup> to I<sub>2</sub>. Add 1 mL of hexane and agitate the mixture. A purple color of  $I_2$  in the hexane layer indicates  $I^-$  was present in the original sample.

$$2 I^{-}(aq) + 2 Fe^{+3}(aq) \rightarrow I_{2}(aq) + 2Fe^{+2}(aq)$$
 (4)

- 5. Test for the Phosphate Ion
  - (A) If no I<sup>-</sup> was present, mix about 2 drops of 0.5 M (NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub> reagent with 5 drops of 6 M HNO<sub>3</sub> to 1 mL of a new test solution.
  - (B) If I<sup>-</sup> was present, add 5 drops of 6 M HNO<sub>3</sub> to 1 mL of a new test solution and boil the test tube for 5 to 10 minutes to remove the iodide. Then add 2 drops of the ammonium molybdate reagent to the test solution.

A yellow precipitate of ammonium phosphomolybdate, (NH<sub>4</sub>)<sub>3</sub>PO<sub>4</sub>·12MoO<sub>3</sub>, appearing at once or after the mixture has been warmed a few minutes to 40 °C indicates the presence of  $PO_4^{-3}$ .

$$21NH_{4}^{+}(aq) + 12[Mo_{7}O_{24}^{-6}](aq) + 7H_{3}PO_{4}(aq) + 51H^{+}(aq) \rightarrow 7(NH_{4})_{3}PO_{4} \cdot 12MoO_{3}(aq) + 36 H_{2}O(l)$$
(5)

Record your observations for your known and unknown solutions below. Determine the identity of your unknown.

#### **Experiment Observations and Results:**

UNKNOWN\_\_\_\_\_ IONS PRESENT\_\_\_\_\_

# **Pre-Lab Assignment:**

1. Construct separate flow charts for the identification of the various five anions in a known sample. Refer to Experiment #10 for guidelines on preparing your flow charts.

#### Post-Lab Assignment: Anion Analysis

A solution may contain Cl<sup>-</sup>, CO<sub>3</sub><sup>-2</sup>, PO<sub>4</sub><sup>-3</sup>, and/or SO<sub>4</sub><sup>-2</sup>. No effect is observed upon addition of 6 M HNO<sub>3</sub>; this resulting mixture will be referred to as solution 1. No effect is observed on addition of 0.1 M AgNO<sub>3</sub> to solution 1. A white precipitate is reported on addition of 1 M BaCl<sub>2</sub> to solution 1. Finally, a yellow precipitate is observed on addition of 0.5 M (NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub> to solution 1. Which of the ions are present, which are absent, and which remain undetermined? State your reasoning below. <u>NOTE</u>: simply listing ions below without the appropriate reasoning will NOT earn you any credit!

Present \_\_\_\_\_\_Absent

In Doubt \_\_\_\_\_

### Pre-Lab Assignment: Electrochemical Cells Experiment

Answer each of the following questions and place the responses on the lines provided.

1. The following data were measured using a nickel electrode as the standard:

$\mathrm{Cu}^{2+}(\mathrm{aq}) + 2e^{-} \rightarrow \mathrm{Cu}(\mathrm{s})$	0.62 V
$Ni^{2+}(aq) + 2e^{-} \rightarrow Ni(s)$	0.00 V
$Fe^{2+}(aq) + 2e^{-} \rightarrow Fe(s)$	–0.15 V
$Al^{3+}(aq) + 3e^{-} \rightarrow Al(s)$	-1.38 V

- A. Which ion is most easily reduced?
- B. Which metal is most easily oxidized?
- C. The copper and aluminum electrodes are connected in a battery.
  - 1) Which is the anode? cathode?

Anode: \_\_\_\_\_ Cathode: \_\_\_\_\_

2) Which is oxidized? reduced?

Oxidized: \_\_\_\_\_ Reduced: \_\_\_\_\_

3) What will the battery voltage be?

4) Write a balanced net ionic equation for the reaction that takes place.

# **Experiment**

<u>Part 1A</u>:  $E^{o}$  for: Zn | 1.0 M Zn<sup>+2</sup> | | 1.0 M Cu<sup>+2</sup> | Cu

Theoretical: \_\_\_\_\_ V Experimental: \_\_\_\_\_ V

<u>Part 1B</u>:  $E_{cell}$  for: Zn | 0.10 M Zn<sup>+2</sup> | | 1.0 M Cu<sup>+2</sup> | Cu

Theoretical: \_\_\_\_\_ V Experimental: \_\_\_\_\_ V

<u>Part 1C</u>:  $E_{cell}$  for: Zn | 1.0 M Zn<sup>+2</sup> | | 0.10 M Cu<sup>+2</sup> | Cu

Theoretical: \_\_\_\_\_ V Experimental: \_\_\_\_\_ V

Part 2:  $E^{o}$  and  $\mathbf{K}_{sp}$  for: Cu | Cu(OH)<sub>2</sub> in 1.0 M OH<sup>-</sup> | | 1.0 M Cu<sup>2+</sup> | Cu

Experimental  $E^o =$ \_\_\_\_\_V

Calculate experimental  $\mathbf{K}$ sp using experimental  $\mathbf{E}^{\circ}$  and the Nernst equation at equilibrium.

Experimental K<sub>sp</sub> = \_\_\_\_\_

Calculate the theoretical  $E^{\circ}$  using a literature value for Ksp = 2.2 x 10<sup>-20</sup>.

Theoretical  $E^o =$ \_\_\_\_\_V

Calculate the % error in  $E^{\circ}$ .

% error in  $E^{\circ}$  = \_\_\_\_\_

Part 3:  $E^{o}$  and  $K_{f}$  for: Cu | Cu(NH<sub>3</sub>)<sub>4</sub><sup>2+</sup> in 1.0 M NH<sub>3</sub> | | 1.0 M Cu<sup>2+</sup> | Cu

Experimental  $E^o =$ \_\_\_\_\_V

Calculate experimental  $\mathbf{K}_{\mathbf{f}}$  using experimental  $\mathbf{E}^{o}$  and the Nernst equation at equilibrium.

Experimental K<sub>f</sub> = \_\_\_\_\_

Calculate the theoretical  $E^{\circ}$  using a literature value for  $K_{f} = 1.7 \times 10^{13}$ .

Theoretical  $E^o = \__V$ 

Calculate the % error in  $E^{\circ}$ .

% error in  $E^{\circ}$  = \_\_\_\_\_

## **Post-Lab Questions:**

1. Sketch and label well with movement arrows the processes occurring in the standard Daniell cell (from Part 1A) while it is operating. Note that the electrode reactions produce and consume ions at the electrodes.

2. Briefly comment on how well your experimental cell potentials match the theoretical cell potentials in the standard and nonstandard Daniell cells from parts 1A/1B/1C.

3. How should the voltage change in the Daniell cell if the zinc solution is made more dilute (and the concentration of the copper solution is kept the same)? Explain your choice.

4. Calculate the solubility product of silver chloride, an extremely insoluble salt, from the following oxidation potentials:

 $Ag \rightarrow Ag^{+} + e^{-} \qquad E^{\circ} = -0.7996 V$   $Ag + Cl^{-} \rightarrow AgCl + e^{-} \qquad E^{\circ} = -0.2221 V$ 

5. Construct a standard voltaic cell diagram that will enable you to determine the solubility product of AgCl from the half reactions given in the previous question.

6. A cell that has the following half reactions was constructed.

 $\begin{array}{rcl} Cd^{2+} &+ & 2e^- \rightarrow & Cd \\ Cd &+ & 3 & en & \rightarrow & Cd(en)_3^{2+} &+ & 2e^- \end{array}$ 

The measured  $E^{\circ}$  at 25 °C was +0.38 V. Calculate the experimental K<sub>f</sub>.

## **Pre-Lab Questions**: Determination of an Equivalent Mass by Electrolysis

1. In an electrolysis cell similar to the one employed in this experiment, a student observed that his unknown metal anode lost 0.233 g while a total volume of 94.50 mL of H<sub>2</sub> was being produced. The temperature in the laboratory was 25 °C, and the barometric pressure was 740 mm Hg. At 25 °C, the vapor pressure of water is 23.8 mm Hg. To find the equivalent mass of his metal, the student filled in the blanks below:

$P_{hydrogen gas} = P_{bar} - VP_{wate}$	r =	mm Hg =	atm
V hydrogen gas =	mL =	L	
T = K			
$n_{\text{hydrogen gas}} = $	moles		
1 mol H <sub>2</sub> requires passage	of	faradays	
Faradays passed (moles of	electrons) =		
Loss of mass of metal anot	le =	g	
Grams of metal lost per far	aday passed = grams	s lost/faradays passed =	
			g = GEM
The student was told that the	ne identity of the me	tal anode is copper.	
MM Cu =	_ g. The charge <i>n</i> on	the Cu ion is	(Eq. 3)

# Data and Calculations: Determination of an Equivalent Mass by Electrolysis

Mass of metal anode	g
Mass of anode after first electrolysis	g
Mass of anode after second electrolysis	g
Initial buret reading	mL
Buret reading after first electrolysis	mL
Initial buret reading for the second electrolysis	mL
Buret reading after second electrolysis	mL
Barometric pressure	mmHg
Temperature T	°C
Vapor pressure of $H_2O$ at $T$	mmHg
Total volume of $H_2$ produced, V	mL
Temperature T	K
Pressure exerted by dry H <sub>2</sub> : $P = P_{bar} - VP_{water}$ (ignore any pressure effect due to liquid levels in the buret)	mmHg
Moles of $H_2$ produced, $n$	moles
Faradays passed (moles of electrons)	
Loss in mass by anode	g
Equivalent mass of metal (GEM = g lost / faradays passed)	g
Unknown metal number	
Metal	
ММ	g
Charge <i>n</i> on cation (equation 3)	

## **Pre-Lab Questions**: Determination of an Equivalent Mass by Electrolysis

1. In ordinary units, the faraday is equal to 96,485 coulombs. A coulomb is the amount of electricity passed when a current of one ampere flows for one second. Given the charge on an electron,  $1.6022 \times 10^{-19}$  coulombs, calculate a value for Avogadro's number.

2. Consider the electrolysis of Na<sub>2</sub>SO<sub>4</sub>(aq). Write the overall net ionic equation that occurs for this electrolysis. Note: Consider all the possible reactions; the oxidation of water or sulfate ions, the reduction of water or sodium ions. Determine which is more likely to occur.

\_\_\_\_\_

#### Data

Weight of salicylic acid added	
Volume of acetic anhydride	
Density of acetic anhydride	
Molecular Weight of acetic anhydride	
Molecular Weight of salicylic acid	
Theoretical Yield of aspirin	
Actual Yield of crude aspirin	
Actual Yield of recrystallized aspirin	
Percent Yield of recrystallized aspirin	
Melting Point of pure aspirin (literature)	
Melting Point of recrystallized aspirin	
weiting I offic of reerystanized aspiriti	

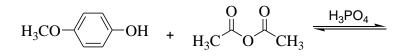
#### **Discussion Questions:**

- 1. Determine the percentage yield of your crude product.
- 2. As in many organic reactions, the synthesis in this experiment is an equilibrium reaction. What steps could you take to improve the yield of aspirin in this particular experiment?

3. If the aspirin crystals were not completely dried before the melting point was determined, what effect would this have on the observed melting point?

4. *(Optional)* Discuss the purity of your final product. How pure (or impure) is your aspirin based on literature values? Comment on how your IR spectrum parallels the spectrum of pure acetylsalicylic acid, making certain to LABEL and discuss all characteristic absorption peaks. You should turn in the labeled IR spectrum of your product with this report.

5. Consider the reaction shown below. Predict the product(s) of this reaction.



Post-Lab Questions: Synthesis and Characterization of Aspirin

1. Determine the theoretical yield of aspirin that can be obtained from the addition of 2.0 grams of salicylic acid and 5.0 mL of acetic anhydride (density = 1.08 g/mL). What is the limiting reagent?

2. Determine the percentage yield of the reaction if 1.9 g of aspirin is obtained in this experiment.

3. What is the purpose of recrystallization?

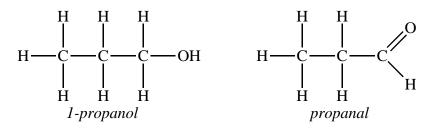
4. Draw and name two different structural isomers for esters (RCOOR) with an empirical formula  $C_3H_6O_2$ .

# Part B Liquid Unknowns

Name of Solvent	Formula	Draw the 3D structure
acetone or 2-propanone	C3H6O	
ethyl acetate or ethyl ethanoate	C4H8O2	
methanol	CH₄O	
ethanol	C2H6O	
1-propanol	C3H8O	
2-propanol or isopropanol	C3H8O	
methyl acetate or methyl ethanoate	C3H6O2	
methyl ethyl ketone or 2-butanone	C₄H8O	

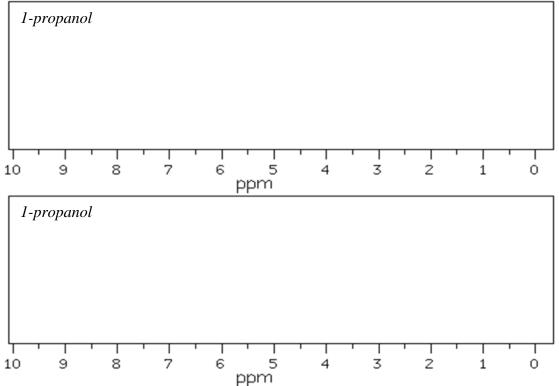
## Post-Lab Questions: Introduction to IR and NMR

Consider the following two molecules:



1. What should you see in their infrared spectra that should allow them to be distinguished?

- 2. Label each of the hydrogen atoms in the molecules above with the letters "a," "b," "c," etc. to indicate all of the different types of hydrogen.
- 3. For each of the molecules, draw what you would expect the <sup>1</sup>H NMR spectrum to look like. Label each signal with the letters that you used in question 2 ("a," "b," "c," etc.). Be sure to place the signals in the appropriate chemical shift order and to draw *and label* the appropriate splitting pattern:



# **Problem Set**

1. A student performs an experiment to calculate the specific heat capacity of copper. The student experimentally finds the answer 0.340 J/g°C. Looking up the accurate published value it is found to be 0.385 J/g°C. Solve for the student's percent error.

Percent error =  $\left| \frac{\text{Experimental} - \text{True}}{\text{True}} \right| \times 100 \%$ 

2. Since 1965, dimes are composed of copper with 25% nickel on the outside. A Roosevelt Type dime (1946 to 1964), designed by John R Sinnockis, is composed of 90.0% silver and 10.0% copper. The composition changed when the dime cost more in silver than it was worth. A 1963 dime is weighed on ten different balances, and the mass is recorded.

Balance Number	Mass $(g) = x_m$	$\mathbf{d} = \mathbf{x}_{\mathbf{m}} - \overline{\mathbf{x}}$	d <sup>2</sup>
1	2.495 g		
2	2.509 g		
3	2.507 g		
4	2.511 g		
5	2.508 g		
6	2.538 g		
7	2.512 g		
8	2.501 g		
9	2.510 g		
10	2.490 g		

- a) Solve for the average value,  $\overline{\mathbf{x}}$ :
- b) Fill in the chart for all the d and  $d^2$  values.

c) Solve for the standard deviation, s.

$$s = \sqrt{\frac{\sum d^2}{n-1}}$$

- d) Solve for the range,  $\bar{\mathbf{x}} \pm 2s$ .
- e) Check all data points against the range. Identify values outside the range that may be unreliable and discarded:
- f) Do you suspect the differing values are due to random errors or systematic errors or possibly both? Explain. How might you test your hypothesis?

g) Solve for the new average value,  $\bar{x}$ , removing values outside the range in part (e).

Note: Once unreliable data points are discarded the process should be repeated to recalculate  $\bar{x}$ , d, s, and range values. Only the recalculation of  $\bar{x}$  is required for the problem set today, but you are encouraged to recalculate d, s and range on your own.

3a) A student determines the concentration of a sodium hydroxide solution by titration with standardized KHP. S/he obtains the values: 0.190 M, 0.202 M, and 0.205 M. Should the value 0.190 M be rejected? Apply the *Q Test*. For three values Q must be greater than 0.94 to reject the number.

 $Q = \left| \frac{suspect-nearest}{largest-smallest} \right|$ 

b) The student decides to repeat the experiment two more times. The five values now include: 0.190 M, 0.202 M, 0.205 M, 0.201M and 0.203M. Use the *Q Test* to see if the first value may be rejected. For five values Q must be greater than 0.64 to reject the number.

c) Solve for the average Molarity of the measurements from part b with and without the rejected number. Is there value in repeating an experiment several times?

4. (Take home assignment). A set of solution densities as a function of weight/volume % sugar is given below. Note that weight/volume % sugar refers to how many grams of sugar per 100 mL of solution. As an example, 9.000 % means that there are 9.000 g of sugar per 100 mL of solution. Use Excel<sup>®</sup> (or similar program) to construct a density (y-axis) versus weight/volume % sugar (x-axis) plot. Add a linear fit through the Add Trendline function and display the equation and the r<sup>2</sup> value on your chart. Examine your r<sup>2</sup> value and your plot. You will notice, upon visual inspection, that there are four data points that can be considered outliers. Remove these data points, one set at a time by highlighting and then deleting the x,y values on the columns. As you delete the outliers, one data set at a time, you will see that the graph, the equation and the r<sup>2</sup> change accordingly. Note how the r<sup>2</sup> value changes. By the last deletion, you will now have an r<sup>2</sup> value that is generally acceptable.

Print out two graphs, (1) use all data points, (2) without all four outliers, and submit to your instructor in lab next week. Graphs should conform to the five guidelines given in the introduction.

weight/volume % sugar	density of solution (g/mL)
0.00	0.998
2.007	1.017
3.070	1.002
4.000	1.009
5.010	1.008
6.094	1.036
6.991	1.017
8.008	1.020
9.000	1.028
10.00	1.030
11.12	1.033
12.11	1.053
13.01	1.041
15.00	1.050
16.00	1.055
17.02	1.055
18.00	1.056
19.00	1.060
21.03	1.071
23.05	1.066
24.02	1.080

Display the equation and  $r^2$  value.