CHE116

Experiments and Worksheets Part 2: E & W 8 – 13, and a Review

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Experiment 8: The Heat of Neutralization

(This experiment was adapted from CC-BY Torres & González-Urbina, CUNY)

Purpose

This experiment introduces the technique of calorimetry. The enthalpy change of acid base neutralization reactions will be determined using a coffee-cup calorimeter. The heat released or consumed by the reactions will allow for the calculation of the enthalpy change, ΔH . The chemical reactions will be (1) HCl with NaOH, and (2) HCl with NH₃, and (3) NH₄Cl with NaOH.

Background

Reactants and products in a chemical reaction normally have different amounts of energy, and this difference in energy is reflected by a release or absorption of heat during the reaction. By means of a coffee-cup calorimeter and a thermometer, the temperature changes produced by a chemical reaction can be measured. When a reaction produces heat, the temperature inside the calorimeter will rise. When a reaction consumes heat, the temperature inside the calorimeter will decrease. These heat (energy) changes are related to the enthalpy change of the chemical reaction. This is a classical college chemistry experiment implemented in many lab manuals with different choices of acids and bases [1-4].

Measurement of heat

Heat (thermal energy, q) is the energy transferred as a result of a difference in temperature. Heat flows from regions of high-temperature to low-temperature. Heat is connected to the change of temperature, ΔT , by means of the specific heat C: $q = m \cdot C \cdot \Delta T$ where m is the mass of the object and ($\Delta T = T_{final} - T_{initial}$). For aqueous solutions, the specific heat of water is used, 4.184 J · (°C⁻¹ · g⁻¹),

Enthalpy change of a chemical reaction

The energy change in a chemical reaction measured at constant pressure (in an open container) is called the enthalpy change, ΔH . All chemical reactions are accompanied by a change in energy. For example, for the case of HCl and NaOH is the following reaction:

HCl + NaOH \rightarrow NaCl + H₂O Δ H = -52 kJ / mole

The heat involved in this process is -52 kJ / mole (per mole means with the coefficients shown in the balanced equation). The negative sign means that the heat is being released. Reactions that release heat are called exothermic ($\Delta H < 0$), whereas reactions that absorb heat are called endothermic ($\Delta H > 0$).

Using a coffee-cup calorimeter

Two nested polystyrene coffee-cups make a good constant-pressure calorimeter, as polystyrene is a good heat insulator. With a coffee-cup calorimeter and a thermometer, we can measure the enthalpy change, ΔH , of a chemical reaction happening inside the calorimeter: the heat, q, consumed or released inside a calorimeter is related to the enthalpy change of the reaction by: $\Delta H_{rxn} + q = 0$ which rearranges to $\Delta H_{rxn} = -q$

Often times heat flows through the walls of calorimeter, as these devices are never perfect. To take this loss of heat into account, we can use the following formula:

 $\Delta H_{rxn} = (-1) \cdot ((m \cdot C \cdot \Delta T) + (C_{Cal} \cdot \Delta T)) \qquad \text{where } C_{Cal} \text{ is the calorimeter constant and it is calculated to be } C_{Cal} = 10.0 \text{ J} \cdot ^{\circ} \text{C}^{-1} \text{ for a standard calorimeter. It accounts for the energy absorbed by the calorimeter.}$

Example Calculation:

When 12.00 ml of 1.0 M, HNO₃, reacts with an of excess NaOH (but no additional volume): $HNO_3(aq) + NaOH(aq) \rightarrow NaNO_3(aq) + H_2O(aq)$ in a coffee-cup calorimeter with a heat capacity of 10. J ·°C⁻¹, the temperature of the calorimeter rises by 9.0°C. Calculate the reaction enthalpy for this process assuming the density of the mixture is 1.00 g/mL.

Answer:

Answer: the heat absorbed by the calorimeter is the heat exchanged during the chemical reaction:

Mass of solution = $(d \cdot V)$ where V is the volume used of (nitric acid + NaOH) (for this example, NaOH did not add volume)

 $\Delta H_{rxn} = (-1) \left((m \cdot C \cdot \Delta T) + (C_{Cal} \cdot \Delta T) \right)$

Plugging all the values:

mass of solution = $(1.00g \cdot mL^{-1} \cdot 12.00mL) = 12.0 g$ $\Delta H_{rxn} = (-1) ((12.0g \cdot 4.184 J \cdot C^{-1} \cdot g^{-1} \cdot 9.0C^{-1}) + (10.J \cdot C^{-1} \cdot 9.0C))$

Solving for ΔH_{rxn} we have -542 J. The number of moles of HNO₃ is: n =0.01200 L× 1.0 mole/L =0.012mol. Therefore, on a per mole basis, the ΔH_{rxn} = -542 J / 0.012 mole = -45167 J/mole, which rounds to -45,000 J/mole or -45 kJ /mole.

Hess's Law

Hess's law states that the total enthalpy change during a chemical reaction is the same whether the reaction results from one step or several steps. For example:

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\begin{array}{ccc} HCl + NaOH \rightarrow NaCl + H_2O & \Delta H_1 \\ and & \\ NH_3 + HCl \rightarrow NH_4Cl & \Delta H_2 \\ or & \\ NaOH + NH_4Cl \rightarrow NaCl + NH_3 + H_2O & \Delta H_{rxn} \end{array}
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E. Ostberg & L. Stewart
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You might have noted there is a relationship among these equations: if you reverse the second equation and add it to the first equation, the third equation is generated. This provided the basis for Hess's law and you will find you can predict the enthalpy change for the third reaction by combining the enthalpy change for the first and second reactions:

 $\Delta H_{rxn} = \Delta H_1 + \Delta H_2$ (after reaction 2 is reversed) You will calculate the expected ΔH_{rxn} with your experimentally determined ΔH_1 and ΔH_2 values.

Chemicals

HCl_(aq), NH_{3(aq)}, NaOH_(aq), NH₄Cl_(aq) (Caution: All solutions are 1.0 M concentration.)

(Note: When NH_3 is in solution, it is typically written as $NH_4OH_{(aq)}$. However, the $NH_{3(aq)}$ version is better for the Hess's Law work.)

Equipment

Coffee-cup calorimeter with lid (not air tight, still considered an open container) ONE Thermometer (Use <u>THE SAME</u> thermometer for all temperature measurements.) Graduated cylinders (50 mL or 100 mL) Plastic droppers 150 mL and 250 mL beakers

Procedure (*Make sure to use the same thermometer throughout the experiment so the results are consistent.*) **Measuring** Δ **H for the HCI, NaOH Reaction**

- 1) Obtain a coffee-cup calorimeter with a thermometer and lid.
- 2) Obtain approximately 110 mL of HCl and 110 mL of NaOH, each in their own beaker.
- 3) Place 50.0 mL of HCl and 50.0 ml of NaOH in separate graduated cylinders.
- 4) Measure the temperature of each of these solutions. Make sure you clean the thermometer in between measurements as the chemicals will react. Record the mean of the two temperatures as your initial temperature, ti.
- 5) Get the timer ready.
- 6) Add the acid to the calorimeter. Then add the base. Start the timer immediately and place the lid on the calorimeter. Use the stirrer gently to mix the solutions.
- Record the maximum temperature observed over a 4-minute time period. Do not stop the timer during the trial. Record the maximum temperature (which is the final temperature, t_f).
- 8) Calculate the change of enthalpy, ΔH , by using the following formulas: Mass of solution = (d ·V) where V is the volume of (acid + base) used. $\Delta H_1 = (-1) \left((m \cdot C \cdot \Delta T) + (C_{Cal} \cdot \Delta T) \right)$
- 9) Repeat steps 1-8 for trial 2. Calculate the ΔH for each trial as per mole of acid. Then calculate the average ΔH (use units of J / mole for the average value).

Measuring ΔH for the HCI, NH₃ Reaction

- 1) Clean and dry the coffee cup used previously.
- 2) Obtain approximately 110 mL of HCl and 110 mL of NH₃, each in their own beaker.
- 3) Place 50.0 mL of HCl and 50.0 ml of NH₃ in separate graduated cylinders.
- Measure the temperature of each of these solutions. Record the mean of the two temperatures as your initial temperature t_i.
- 5) Get the timer ready.
- 6) Add the acid to the calorimeter. Then add the base. Start the timer immediately and place the lid on the calorimeter. Use the stirrer gently to mix the solutions.
- Record the maximum temperature observed over a 4-minute time period. Do not stop the timer during the trial. Record the maximum temperature (which is the final temperature, t_f).
- 8) Calculate the change of enthalpy, ΔH , by using the following formulas: Mass of solution = (d ·V) where V is the volume of (acid + base) used. $\Delta H_2 = (-1) \left((m \cdot C \cdot \Delta T) + (C_{Cal} \cdot \Delta T) \right)$
- 9) Repeat steps 1-8 for trial 2. Calculate the ΔH for each trial as per mole of acid. Then calculate the average ΔH (use units of J / mole for the average value).

Measuring ΔH for the NH₄Cl, NaOH Reaction

- 1) Clean and dry the coffee cup used previously.
- 2) Obtain approximately 110 mL of NH₄Cl and 110 mL of NaOH, each in their own beaker.
- 3) Place 50.0 mL of NH₄Cl and 50.0 ml of NaOH in separate graduated cylinders. Make sure the cylinders are clean and dry.
- Measure the temperature of each of these solutions. Record the mean of the two temperatures as your initial temperature t_i.
- 5) Get the timer ready.
- 6) Add the acid to the calorimeter. Then add the base. Start the timer immediately and place the lid on the calorimeter. Use the stirrer to gently mix the solutions.
- Record the maximum temperature observed over a 4-minute time period. Do not stop the timer during the trial. Record the maximum temperature (which is the final temperature, t_f).
- 8) Calculate the change of enthalpy, ΔH, by using the following formulas: Mass of solution = (d ·V) where V is the volume of (acid + base) used. ΔH_{rxn} = (-1) ((m ·C ·ΔT) +(C_{Cal} ·ΔT))
- 9) Repeat steps 1-8 for trial 2. Calculate the ΔH for each trial as per mole of acid. Then calculate the average ΔH (use units of J / mole for the average value).

Name: RCBC CHE116 Notebook Worksheet f	or Experiment 8: The Heat of Neutralization
Section 1 Experiment work was performed on Lab Partner:	
Section 2 Raw Data Measuring ΔH for the HCI, NaOH Reaction Trial 1	on
Volume of 1 M HCl used:	Temp. of HCI:
Volume of 1 M NaOH used:	Temp. of NaOH:
Maximum temperature of mixture during the	e 4 minutes:
Trial 2	
Volume of 1 M HCl used:	Temp. of HCI:
Volume of 1 M NaOH used:	Temp. of NaOH:
Maximum temperature of mixture during the	e 4 minutes:
Measuring ∆H for the HCl, NH₃ (as NH₄O Trial 1	PH) Reaction
Volume of 1 M HCl used:	Temp. of HCI:
Volume of 1 M NH₄OH used:	Temp. of NH₄OH:
Maximum temperature of mixture during the	e 4 minutes:
Trial 2	
Volume of 1 M HCl used:	Temp. of HCI:
Volume of 1 M NH ₄ OH used:	Temp. of NH₄OH:
Maximum temperature of mixture during the	e 4 minutes:
Measuring ΔH for the NH₄CI, NaOH Read Trial 1	ction
Volume of 1 M NH4Cl used:	Temp. of NH₄CI:
Volume of 1 M NaOH used:	Temp. of NaOH:
Maximum temperature of mixture during the	e 4 minutes:
Trial 2	
Volume of 1 M NH ₄ Cl used:	Temp. of NH₄CI:
Volume of 1 M NaOH used:	Temp. of NaOH:
Maximum temperature of mixture during the	e 4 minutes:

Section 3: Experimental Procedure

A calorimetry experiment was performed with the use of nested Styrofoam coffee cups, a thermometer, a stirring loop and a cover. For each trial, the appropriate acid was put into the Styrofoam cup. Then the appropriate base was added. The calorimeter was covered immediately after the base was added, the contents were stirred with the cover on, and the maximum temperature of the calorimeter contents was recorded. The temperature was monitored for four minutes, from the start of the base addition. Each acid, base reaction was done twice, recorded as trial 1 and trial 2. The acid, base combinations used and the volume of each <u>for each trial</u> were: acid, base ______, volume of <u>acid</u> used per trial: ______

acid,base,	volume of	f <u>acid</u> used	d per trial:	

acid,base_____, volume of acid used per trial: _____

Section 4: Data Tables Acid, Base Combination: HCI, NaOH

	Trial 1	Trial 2
Total volume of acid+base used		
Total mass of acid+base		
(assume d=1.00 g/mL)		
Initial temperature of acid+base, Ti		
(average of acid, base temps.)		
Final temperature of mixture, Tf		
(maximum temp. during the 4 minutes)		
$\Delta T = T_f - T_i$		

Acid, Base Combination: HCI, NH₃ (as NH₄OH)

	Trial 1	Trial 2
Total volume of acid+base used		
Total mass of acid+base (assume d=1.00 g/mL)		
Initial temperature of acid+base, Ti (average of acid, base temps.)		
Final temperature of mixture, T _f (maximum temp. during the 4 minutes)		
$\Delta T = T_f - T_i$		

Acid, Base Combination: NH₄CI, NaOH

	Trial 1	Trial 2
Total volume of acid+base used		
Total mass of acid+base		
(assume d=1.00 g/mL)		
Initial temperature of acid+base, Ti		
(average of acid, base temps.)		
Final temperature of mixture, T _f		
(maximum temp. during the 4 minutes)		
$\Delta T = T_f - T_i$		

Chemical reactions used:

HCI + NaOH	\rightarrow	
HCI + NH₃	\rightarrow	
NH₄CI + NaOH	\rightarrow	

Section 5: Calculations & Results Acid, Base Combination: HCI, NaOH

Show your calculations for Trial 1 here: (you do	on't have to show calculations for Trial 2)
Mass of total mixture =	Average t _i =
$\Delta H_1 = (-1) \cdot ((m \cdot C \cdot \Delta T) + (C_{Cal} \cdot \Delta T)) (Th)$	nis will give ΔH in units of Joules.)
Moles acid = M x L	
ΔH_1 / moles acid (This will give ΔH in uni	its of J/mole)
Average ΛH_1 (using the l/mole acid results of Tr	ial 1 and Trial 2)
1	

Show your calculated results here:

$C = 4.184 \text{ J/(°C·g)}$ $C_{cal} = 10.0 \text{ J/C}$	Trial 1	Trial 2
ΔH_1 in units of J		
Moles of acid used		
ΔH_1 in units of J/mole of acid		
Average ΔH_1 in J/mole of acid		

Acid, Base Combination: HCI, NH₃

Mass of total mixture =	Average t _i =	
$\Delta H_2 = (-1) \cdot ((m \cdot C \cdot \Delta T) + (C_{Cal} \cdot \Delta T))$	$(This will give \Delta H in$	units of Joules.)
Moles acid = M x L		
ΔH_2 / moles acid (This will give 2	ΔH in units of J/mole)	
Average ΔH_2 (using the J/mole acid res	ults of Trial 1 and Trial 2)	
Show your calculated results here:		
$C = 4.184 \text{ J/(°C·g)}$ $C_{cal} = 10.0 \text{ J/C}$	Trial 1	Trial 2
ΔH_2 in units of J		
Moles of acid used		
AH ₂ in units of J/mole of acid		
Acid, Base Combination: NH ₄ CI, Na	OH	
Show your calculations for Trial 1 here:	: (you don't have to show	calculations for Trial 2
Mass of total mixture =	Average t _i =	
$\Delta H_{rxn} = (-1) \cdot ((m \cdot C \cdot \Delta T) + (C_{Cal} \cdot \Delta T))$	$\Delta T)$) (This will give ΔH in	units of Joules.)
Moles acid = M x L		
ΔH_{rxn} / moles acid (This will give	e ΔH in units of J/mole)	
Average ΔH_{rxn} (using the J/mole results	of Trial 1 and Trial 2)	
Show your calculated results here:		
$C = 4.184 \text{ J/(°C·g)}$ $C_{cal} = 10.0 \text{ J/C}$	Trial 1	Trial 2
ΔH_{rxn} in units of J		
ΔH _{rxn} in units of J Moles of acid used		
$\begin{array}{l} \Delta H_{rxn} \text{ in units of J} \\ \text{Moles of acid used} \\ \Delta H_{rxn} \text{ in units of J/mole of acid} \end{array}$		

 $HCI + NaOH \rightarrow NaCI + H_2O$

Hess's Law

Calculation ΔH_{rxn} for $NH_4CI + NaOH \rightarrow NaCI + NH_3 + H_2O$

Using your calculated results for:

 $\Delta H_{1average} =$ _____

 $HCI + NH_3 \rightarrow NH_4CI$

 $\Delta H_{2average} =$ _____

Show your Hess's Law calculations here. Show the addition of the chemical equations and the math.

Section 6: Conclusion

Were you able to obtain results for all three reactions? Did your $\Delta H_{1average}$ result agree with the expected -52 kJ/mole? (See the bottom of page 3 in the experiment document.) Did your calculated Hess's Law ΔH_{rxn} result agree with your experimental ΔH_{rxn} result? What does this imply about the accuracy of your results? Explain your answers.

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Experiment 9: Titration of Acids

(This experiment was adapted from Titration of a Weak Acid, by CC-BY Torres & González-Urbina, CUNY)

Purpose

Weak acids are acids that do not dissociate completely when dissolved in water, releasing only some of its hydrogen atoms into the solution. Acetic acid (CH₃COOH) is an important weak acid, produced from the fermentation of ethanol in wine. Commercial vinegar is just an aqueous solution of acetic acid. The goal of this experiment is to calculate the molar concentration and %wt of a sample of acetic acid in vinegar by means of a chemical procedure known as titration. You will use your results as a quality control check for the advertised %wt concentration on the bottle of vinegar. In order to do that you will react the acetic acid with a solution of sodium hydroxide (NaOH), which has a known concentration. You will use phenolphthalein as the indicator. A strong acid, HCl, ionizes completely when dissolved in water. The HCl solution of known concentration will be used to first practice the technique of titration, before analyzing the vinegar sample.

Background

A titration is a technique where a solution of known concentration (often times a base) is used to determine the unknown concentration of another solution (often times an acid) [1]. Both substances react with each other in an acid-base reaction. The solution of known concentration is delivered carefully from a buret until an indicator changes color. This experiment is a classic practice, previously implemented in numerous college chemistry laboratory manuals [2-5].

Volumetric Analysis

The determination of concentration by measuring volumes is called volumetric analysis. Titrations are volumetric analyses where a buret is used to add and measure the volume of one of the reactants. In an acid-base titration, an acid reacts with a base by gradually adding one solution to the other. The volume of the acid solution (in the Erlenmeyer flask) is known from the pipet, and the volume of the NaOH solution required for the complete reaction is measured (from the buret).

An indicator is used to indicate the exact end of the reaction. The indicator chosen will have one color before the reaction is complete and a different color when the acid-base reaction finishes. For example, in the reaction between acetic acid (CH₃COOH) and sodium hydroxide (NaOH):

 $CH_3COOH + NaOH \rightarrow CH_3COONa + H_2O$

using phenolphthalein as the indicator, the solution will be colorless before completion of this reaction but pink after completion. At a specific point during the titration, a partial drop of the NaOH from the buret will cause the solution being titrated to turn from colorless to a barely discernible pink color. This point is called the endpoint. The endpoint signals the exact point when the moles H¹⁺ equals the moles of OH¹⁻, which is referred to as the equivalence point.



Figure 13: (Left panel) A buret employed in a titration. (Center panel) Illustration showing the correct way to control the buret valve. (Right panel) The titration set up

References

- (1) Crouch, D. S.D.W.F.L.H. S., Fundamentals of analytical chemistry; Nelson Education: 2013.
- (2) Beran, J. A., Laboratory manual for principles of general chemistry; John Wiley and Sons: 2010.
- (3) Ebbing, R. W.D. D., Experiments in General Chemistry; Houghton Mifflin Harcourt Publishing Company: 2004.
- (4) Russo, T., Merrill Chemistry Lab Manual; McGraw-Hill: 1998.
- (5) Holman, G. H. J., Chemistry in Context; McGraw-Hill Education: 2014.

Chemicals

0.075 M HCl solution 0.10 M NaOH solution Commercial vinegar (acetic acid solution) Phenolphthalein 1% solution

Equipment

Buret setup, (50 mL buret) Erlenmeyer flask, 125 mL Plastic droppers Volumetric flask, 100 mL Pipet, 10 mL, and pipettor Funnel Clean, dry beakers (various sizes)

Procedure

Set-Up

- 1) Obtain a 10 mL pipet and pipettor, a 50 mL buret, a buret stand, and buret clamp.
- 2) Obtain about 50 mL of hydrochloric acid solution in a clean, dry 100 mL beaker and about 80 mL of the NaOH solution in a clean, dry 150 mL beaker.
- Rinse your buret with deionized water. Use a funnel to rinse your buret three times with approximately 2 mL of NaOH solution per rinse, and then fill it with the NaOH solution.
- 4) Remember to drain the NaOH solution so the meniscus is within the calibration marks and the air in the tip is pushed out.

Doing the titrations

Hydrochloric Acid (To prove you can do a titration.)

- Pipet 20.0 mL of hydrochloric acid into a clean 125 mL Erlenmeyer flask. Add approximately 10 mL of distilled water and 3 drops of phenolphthalein.
- 2) Place the flask under the buret.
- 3) Record the initial volume in the buret. <u>Record all buret volumes with</u> <u>two digits after the decimal point.</u>
- 4) Add the NaOH solution from the buret to the Erlenmeyer flask, while swirling the solution in the flask. Add the NaOH fast in the beginning. As you approach the endpoint, slow the addition of NaOH. By the time you get close to the endpoint, you should be adding the NaOH dropwise, and then by partial drops.
- 5) The titration is completed when the addition of a partial drop of NaOH causes the color to change from colorless to a very light shade of pink.
- 6) Record the final buret volume.
- 7) Repeat the steps above until you have 3 useable trials. A useable trial is one in which the final color in the flask was light pink.
- 8) Calculate the concentration of the original hydrochloric acid solution, for each of the three trials. Calculate the standard deviation for these three trials.

Vinegar

- Vinegar is advertised as 5%wt. acetic acid (this is 0.8 M). The vinegar is too concentrated for a titration using 0.10 M NaOH. You will need to dilute the vinegar. Pipet 10.0 mL of the 0.8 M vinegar solution into the 100.0 mL volumetric flask. Dilute to volume with DI water. Calculate the molarity of this new, diluted solution of vinegar.
- Pipet 20.0 mL of the <u>diluted</u> vinegar into a clean 125 mL Erlenmeyer flask. Add approximately 10 mL of distilled water and 3 drops of phenolphthalein. Swirl to mix.
- 3) Place the flask under the buret.
- 4) Record the initial volume in the buret. <u>Record all buret volumes with</u> <u>two digits after the decimal point.</u>
- 5) Add the NaOH solution from the buret to the Erlenmeyer flask, while swirling the solution in the flask. Add the NaOH fast in the beginning. As you approach the endpoint, slow the addition of NaOH. By the time you get close to the endpoint, you should be adding the NaOH dropwise, then by partial drops.
- 6) The titration is completed when the addition of a partial drop of NaOH causes the color to change from colorless to a very light shade of pink that persists for at least one minute.
- 7) Record the final buret volume.
- 8) Repeat steps 2 6 above two more times, to get 3 useable trials; these three titrations of the diluted vinegar will be used for the calculations.

- 9) Calculate the acetic acid molarity concentration in the diluted vinegar solution, in each of the three trials, and then the average acetic acid molarity. Calculate the standard deviation for these three trials.
- 10)Calculate the average molarity of acetic acid in the original vinegar solution, before the dilution. (Use the diluted average value for this calculation.)
- 11) Use the average acetic acid molarity in the original vinegar solution to calculate the %wt acetic acid in the original vinegar solution. Assume the density of the vinegar is 1.0 g/mL.

Is the vinegar solution really 5% acetic acid as advertised on the label?

The steps and equation for calculating the standard deviation for the three trials of HCI and then the three trials of diluted vinegar are:

Step 1: Calculate the average of the trials

Step 2: Subtract the average from each trial, to get the deviation. Use the absolute value, (+).

Step 3: Square each deviation.

Step 4: Add the squared deviations together.

Step 5: Divided the answer from step 4 by the (number of trials - 1)

Step 6: Take the square root of the answer from step 5.

 $\frac{((\text{dev. 1})^2 + (\text{dev. 2})^2 + (\text{dev. 3})^2)}{(3 - 1)}$

deviation 1 = / trial 1 - average /
deviation 2 = / trial 2 - average /
deviation 3 = / trial 3 - average /

Name: RCBC CHE116 Notebook Worksheet for I Section 1 Experiment work was performed on Lab Partner:	Experiment 9: Titration of Acids
Section 2 Raw Data Titration of HCI Trial 1: Volume of HCI put into Erlenmeyer flask:	Iume of phenolthalein added: drops
Initial buret volume NaOH:, Fin	al buret volume NaOH:
Volume of HCI put into Erienmeyer flask: Volume of DI water added:, Vo Initial buret volume NaOH:, Fin	lume of phenolthalein added:drops al buret volume NaOH:
Trial 3: Volume of HCI put into Erlenmeyer flask: Volume of DI water added:, Vo Initial buret volume NaOH:, Fin	lume of phenolthalein added: :drops al buret volume NaOH:
Titration of Vinegar (the acetic acid) Dilution of the vinegar: Volume of vinegar pipetted into volumetric flas Total volume of the diluted vinegar (size of flas	sk:sk):
Trial 1: Volume of diluted vinegar put into the Erlenme Volume of DI water added:, Vo Initial buret volume NaOH:, Fin	eyer flask: lume of phenolthalein added:drops al buret volume NaOH:
Trial 2: Volume of diluted vinegar put into the Erlenme Volume of DI water added:, Vo Initial buret volume NaOH:, Fin	eyer flask: lume of phenolthalein added:drops al buret volume NaOH:
Trial 3: Volume of diluted vinegar put into the Erlenme Volume of DI water added:, Vo Initial buret volume NaOH:, Fin	eyer flask: lume of phenolthalein added:drops al buret volume NaOH:

Section 3: Experimental Procedure

A _____mL buret was cleaned and filled with _____M NaOH solution. ____mL of the HCl solution was put into a ____mL Erlenmeyer flask. _____mL of DI water was added to the flask, along with _____drops of phenolthalein indicator. The contents of the flask were titrated with the NaOH solution in the buret, until the _____ colored endpoint was reached. These titration steps were repeated _____ more times to obtain three useable titration trials.

A sample of vinegar was diluted: _____ mL of vinegar was pipetted into a _____mL volumetric flask, and the flask was filled to the calibration mark with DI water. ____mL of the diluted vinegar solution was put into a ____mL Erlenmeyer flask. _____mL of DI water was added to the flask, along with _____drops of phenolthalein indicator. The contents of the flask were titrated with the NaOH solution in the buret, until the _____ colored endpoint was reached. This was trial 1. These titration steps were repeated _____ more times to obtain three usable titration trials.

The calculations for the HCl titration and the vinegar titration were then done.

	Trial 1	Trial 2	Trial 3
Volume of HCI			
used			
Initial vol NaOH			
Final vol. NaOH			
Vol NaOH used			

Section 4: Data Tables

	Trial 1	Trial 2	Trial 3
Vol. of diluted vinegar used			
Initial vol. NaOH			
Final vol. NaOH			
Vol. NaOH used			

Section 5: Calculations & Results

For the titration of HCI

Moles NaOH used, Trial 1:

Molarity HCI, Trial 1:

Average Molarity HCI:

Standard Deviation:

For the titration of HCl, show your calculated results here:

	Trial 1	Trial 2	Trial 3
Moles NaOH used (M x L = moles)			
Moles HCl (same as NaOH)			
Vol. HCl used			
Molarity HCl (moles / L = M)			
	•		•

Standard Deviation:

For the titration of diluted vinegar

Show your calculations in this table as indicated

Moles NaOH used, Trial 1:

Molarity acetic acid in diluted vinegar, Trial 1:

Average Molarity acetic acid in diluted vinegar:

Standard Deviation:

For the titration of diluted vinegar, show your calculated results here:

	Trial 1	Trial 2	Trial 3	
Moles NaOH used (M x L = moles)				
Moles acetic acid in diluted vinegar (same as NaOH)				
Vol. diluted vinegar used				
Molarity acetic acid in diluted vinegar (moles / L = M)				
Average Molarity Acetic A	Acid in Dil. Vinegar	:	Standard De	viation = _

For the commercial vinegar calculations:

Show your calculations here			
Molarity of Acetic Acid in Original Vinegar (before dilution):			
% Wt. Acetic Acid in Vinegar:			

Commercial Vinegar calculated results (using the average molarity acetic acid)

Average Molarity of Acetic Acid in Dil. Vinegar	
Molarity of Original Vinegar (before dilution)	
% Wt. Acetic Acid in Vinegar	

Section 6: Conclusion

The molarity of HCI was known; it was 0.075 M. What molarity did you calculate for your experimental result? What can you say about your titration accuracy? Explain your answers.

The vinegar manufacturer claims a 5 %wt acetic acid concentration. What concentration did you calculate? Based on the accuracy of your HCl titrations, what can you say about the actual concentration of acetic acid in the commercial vinegar? Explain your answer.

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Experiment 10: The Measurement and Calculation of pH

Purpose

The purpose of this experiment is to introduce students to the concept of pH values. This also involves the measurement of pH values and calculation of pH values. Various aqueous solutions will be used.

Background

Aqueous solutions are often described as being acidic, basic, or neutral. These terms refer to the concentration of hydrogen ion in solution (written as $[H^{1+}]$ or $[H_3O^{1+}]$). When providing numerical values for these acidic, basic, or neutral solutions, a pH value is typically provided. It is easier for the general public to say and understand a value of pH = 5.00 rather than $[H^{1+}] = 1.0 \times 10^{-5}$ M. People learn the pH scale, and with enough experience, a person gets a feel for what is acidic, basic, and neutral just by hearing a pH value.

The pH scale is typically represented as:



Measuring a pH value can be done in different ways. A pH meter is an instrument that measures the pH of a solution and gives a pH value with one or two significant figures after the decimal point. Indicators are chemicals that change color depending on the pH of their environment. Indicators can be made into an aqueous solution or be put on paper (such as litmus paper). Indicators provide estimated pH values only; they typically do not provide significant figures after the decimal point. For this experiment, both pH meters and indicator solutions will be used to determine the pH of some solutions.



Chemicals

0.0010 M HCI	0.0010 M NaOH	Solution of $pH = 4.0$
0.0010 M CH ₃ COOH	0.0010 M Na ₂ CO ₃	Solution of $pH = 7.0$
Phenolphthalein indicator	Bromothymol Blue indicator	Methyl Orange indicator

Equipment

Beakers, 50 mL, 150 mL	10.0 mL pipet and pipettor	Test tubes
100 mL volumetric flasks	pH meter	Test tube rack
Plastic droppers	Kimwipes	Parafilm

Procedure

Part 1: Estimating the pH of the Original Solutions with Indicators

1. Use four medium size beakers (150 mL) that are clean and dry. Obtain approximately 100 mL of each of these solutions: HCl, CH₃COOH, NaOH, and Na₂CO₃. Bring these solutions back to your lab bench.

2. Obtain 12 medium or large size test tubes. Rinse each test tube well with tap water twice, and then with DI water once. Invert each test tube in the rack, over a piece of paper towel, to drain.

3. Use a plastic dropper to put approximately 2 mL of each solution into three separate test tubes. There will be three test tubes with HCl, three with CH₃COOH, three with NaOH, three with Na₂CO₃. (Refer to Figure 1)

4. Use a dropper bottle of each indicator. There will be one bottle of phenolphthalein, one bottle of bromothymol blue, and one bottle of methyl orange.

5. Put one drop of indicator into the test tubes, as shown in Figure 1 (only one indicator per test tube). Swirl the test tube to mix; there should not be any color-layers. If you see layers, continue to mix. Record the color in each test tube. (If the color is too faint, add one more drop of indicator.)

6. Dispose of the test tube contents into the waste bottle. Rinse each test tube with tap water twice, and then with DI water once. Invert to drain over a piece of paper towel.



Part 2: Diluting the Original Solutions

1. Pipet 10.0 mL of the HCl solution into a 100 mL volumetric flask. Dilute to the mark with Dl water, cover with Parafilm, and invert to mix (invert 50 times).

2. Repeat step 1 for the other original solutions: CH₃COOH, NaOH, Na₂CO₃ SAVE THESE DILUTED SOLUTIONS FOR PARTS 4 & 5

Part 3: Measuring the pH of the Original Solutions with a pH Meter

1. Use the original solutions obtained in Part 1, step 1. (No indicator added.)

2. Use the pH meter according the procedure shown by your instructor (Exp. 10 Bb video). Remember to rinse the electrode with the DI water squirt bottle, then dip the electrode into a beaker with DI water, then **gently** dry the electrode with a Kimwipe. Place the electrode into the original solution of HCl in its beaker. Wait until the reading is stable, and then record the pH value on the display. Record all of the digits on the display.

- 3. Remove the pH electrode from the solution.
- 4. Repeat steps 1 3 for the remaining solutions.

Part 4: Estimating the pH of the Diluted Solutions with Indicators

Repeat Part 1 using the diluted solutions prepared in Part 3.

Part 5: Measuring the pH of the Diluted Solutions with a pH Meter

Repeat Part 3 using the diluted solutions prepared in Part 2.

Dispose of all solutions in the waste bottles in the hoods. Rinse the glassware and test tubes and return to the correct locations.

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Name: _____

Section 1

Experiment work was performed on _______

Section 2 Raw Data

Part 1: Record the color of each <u>original</u> solution with indicator in the table below. The table is arranged according to Figure 1.

Indicator	HCI	CH3COOH	NaOH	Na2CO3
Phenolphthalein				
Bromothymol Blue				
Methyl Orange				

Part 2: No data table is required as long as the original solutions were diluted according to the procedure. If the dilutions were not done as directed, please remake the dilutions according to the procedure.

Part 3: Record the measured pH of each original solution in the table below.

	HCI	CH3COOH	NaOH	Na2CO3
pH meter reading				

Part 4: Record the color of each <u>diluted</u> solution with indicator in the table below. The table is arranged according to Figure 1.

Indicator	HCI	CH3COOH	NaOH	Na2CO3
Phenolphthalein				
Bromothymol Blue				
Methyl Orange				

Part 5:	Record the me	asured pH of	each diluted	solution in the	table below.

	HCI	CH3COOH	NaOH	Na2CO3
pH meter reading				

Section 3: Procedure

Four solutions were obtained from the stock bottles provided. These solutions were referred to as the 'original' solutions: _____, ____,

_____, ____. Approximately _____ mL of each solution were put into three different clean test tubes. One indicator was added to each test tube, following the directions in Table 1. The ______ of each solution was recorded after mixing the contents of each test tube. A pH meter was then used to measure the pH of each original solution (not with the indicator mixed in).

Each original solution was then diluted by pipetting _____ mL into a 100 mL _____ flask, and diluting to the calibration mark with DI water. After mixing well, approximately 2 mL of each diluted solution was put into three different clean test tubes. One indicator was added to each test tube, following the directions in Table 1. The ______ of each indicator was recorded after mixing the contents of each test tube. A pH meter was then used to measure the pH of each diluted solution (not with the indicator mixed in).

Section 4: Data Tables

The tables in this section are arranged differently than in the Raw Data section. This is to help with the comparison of original solutions and diluted solutions.

Indicator	HCI Original, Color	HCI Diluted, Color	CH3COOH Original, Color	CH3COOH Diluted, Color
Phenolphthalein				
Bromothymol Blue				
Methyl Orange				

Indicator	NaOH Original, Color	NaOH Diluted, Color	Na2CO3 Original, Color	Na2CO3 Diluted, Color
Phenolphthalein				
Bromothymol Blue				
Methyl Orange				

	HCI	CH3COOH	NaOH	Na2CO3
Measured pH, Original				
Measured pH, Diluted				

Section 5: Calculations and Results

Parts 1 and 4: Use each indicator color to estimate the pH of the solution. You may only be able to say 'less than' or 'greater than' for some of the colors. (Refer to the image at the bottom of page 23.)

Indicator	HCI Original Estimated pH	HCI Diluted Estimated pH	CH3COOH Orig. Estimated pH	CH3COOH Dil. Estimated pH
Phenolphthalein				
Bromothymol Blue				
Methyl Orange				
OVERALL ESTIMATED pH				

Indicator	NaOH Original Estimated pH	NaOH Diluted Estimated pH	Na2CO3 Orig. Estimated pH	Na2CO3 Diluted Estimated pH
Phenolphthalein				
Bromothymol Blue				
Methyl Orange				
OVERALL ESTIMATED pH				

Parts 3 and 5: For only the HCI solution:

Calculated concentration of H¹⁺ from the measured pH values.

Calculate the $[H^{1+}]$ with the <u>measured pH values</u> as indicated.

	[H ¹⁺] for HCI	[H ¹⁺] = 10 ^{-pH}
Original Soln.		
Diluted Soln.		

Calculated concentration of H¹⁺ from the known molarity of HCI

The [H¹⁺] for the original solution equals the concentration of the original HCl solution.

Calculate the $[H^{1+}]$ of the diluted solution with the (Mc*Vc=Md*Vd) equation.

	[H ¹⁺] for HCI
Original Soln.	
Diluted Soln. use Mc*Vc=Md*Vd	

The concentration of H^{1+} equals the concentration of HCl because there is one H^{1+} for every one HCl, and HCl is a strong acid (100% ionized).

Calculated pH values using the H¹⁺ molarities in the table above.

	pH for HCI
Original Soln.	
Diluted Soln.	

pH = - log [H¹⁺]

Section 6: Conclusion

Parts 3 & 5: Did the diluted solutions have different pH meter values than the original solutions? Why do you think they should have different pH meter values? Explain your answer.

Comment on the pH values obtained with indicators and the pH values obtained with the pH meter. Do you think the indicator pH values are more accurate or less accurate than the pH meter values? Explain your answer.

Experiment 11: Part A: Analysis of Drinking Water by Spectrophotometry Part B: Emission Lines of Elements

Part 1: Purpose

The goal of this experiment is to calculate the concentration of the permanganate ion, MnO₄¹⁻ in solution by means of a spectrophotometer and calibration curve.

Background

Potassium permanganate, KMnO₄, is a strong oxidizing material that can be used for water treatment (https://drinking-water.extension.org/drinking-water-treatment-potassium-permanganate/). When applicable, it is used as a point-of-entry water treatment to control the levels of dissolved iron, manganese, and hydrogen sulfide. It can also be used to control iron bacteria growth in wells. Since potassium permanganate is considered toxic and is harmful to skin, there should not be any excess permanganate left in the drinking water. This experiment will have you analyze a sample of drinking water for the presence of permanganate. You will also be analyzing a quality control sample of known concentration to check if your analysis is accurate.

When light of a specific wavelength passes through a solution containing a substance that absorbs the light, the intensity of the light decreases. The absorption of light can be reported as absorbance, which is measured with a spectrophotometer.

Beer's Law states that the absorbance, A, is directly related to the concentration, c, of the substance that absorbs light (the permanganate ion).

A = abc where a is the molar absorptivity constant specific to a substance, and b is the path length through the sample.

Beer's law allows us to prepare a linear calibration curve, so we can calculate the concentration of permanganate ion in an unknown solute when we know the absorbance by the unknown. The equation of the best-fit line is y = mx + b where y is the absorbance and x is the concentration of the substance (the b here is the y-intercept, not the path length, and m is the slope of the line).

Chemicals

Potassium permanganate stock solution, 5.0 x 10⁻³ M Potassium permanganate quality control sample, 2.5 x 10⁻⁴ M Water sample of unknown permanganate concentration Deionized water

Equipment

Volumetric flasks, 50 mL Test tubes, small 13x100 size Plastic droppers Beakers, assorted small sizes Pipet, 10 mL, and pipettor Test tube rack

Parafilm Spectrophotometer The permanganate ion has an absorbance linear range when the following concentrations are used for the standard solutions (Table 1). Calculate the volume of stock solution needed to prepare each standard solution.

Standard Solution	Concentration, M	Volume of Stock** KMnO ₄
1	1.0 x 10 ⁻⁴	
2	2.0 x 10 ⁻⁴	
3	3.0 x 10 ⁻⁴	
4	4.0 x 10 ⁻⁴	

Table 1

**Stock solution 5.0 x 10⁻³ M, volume of volumetric flask is 50.0 mL.

Procedure

Part A: Preparing the Standard Solutions

- Obtain enough concentrated potassium permanganate solution from the stock bottle in the hood (5.0 x 10⁻³ M). Enough is determined by how much is needed to prepare all of the standard solutions, plus a little extra to avoid getting air bubbles in the pipet.
- 2) Rinse each volumetric flask with deionized water before making the solutions.
- 3) Pipet the appropriate volume of the concentrated solution into each volumetric flask.
- Dilute the contents of each flask with deionized water. Be sure to get the bottom of the meniscus exactly at the calibration line on the neck of each flask. Invert to mix, 50 times.

Part B: Determining the Wavelength for Absorbance

The λ_{max} for the permanganate ion is approximately 530 nm. Test this maximum before finding the absorbance of your solutions.

- 1) Fill a small test tube with standard number 4, to about 2/3 full.
- 2) Fill a small test tube with deionized water, to about 2/3 full. This will be the blank.
- 3) Set the wavelength of the spectrophotometer to 500 nm.
- 4) Put the blank into the sample compartment of the spectrophotometer. Close the lid. Press the 0 ABS button to set the zero mark for the spectrophotometer.
- 5) Remove the blank and put the test tube with standard 5 into the sample compartment. Close the lid. Take the absorbance reading.
- 6) Repeat steps 4 and 5 for the following wavelengths:
 - 510 nm, 520 nm, 525 nm, 530 nm, and 535 nm
- 7) Set the spectrophotometer to the λ_{max} just determined.

Part C: Measuring the Absorbance of the Standard Solutions and Samples

- 1) Use the deionized water blank to re-zero the spectrophotometer at λ_{max} .
- 2) Put standard 1 into a clean, dry test tube, about 2/3 full.
- 3) Wipe the outside of the test tube, and then place it inside the sample compartment of the spectrophotometer.
- 4) Record the absorbance displayed on the instrument readout.
- 5) Repeat steps 2 4 for the remaining standard solutions, the quality controlknown solution (2.5 x 10^{-4} M), and the unknown sample solution.

Part D: Prepare the Calibration Curve and Determine the Concentration of the Samples

- Use Excel or Google Sheets to prepare your calibration curve. This should be a scatter plot, with a linear line. Show the equation of the line on your curve. Also show the R² value. (A perfect calibration curve is when all data points fall on the line; this would have an R² value of 1. The Y-intercept, b, should be zero.)
- Use the standard solution concentrations as the x axis values. Their absorbance values go on the y axis. <u>DO NOT GRAPH</u> the quality control sample and unknown sample.
- 3) Use the equation of the line to calculate the concentration of permanganate ion in the quality control sample and in the unknown sample. The absorbances of these samples are known (the y values). Solve for their concentrations (the x values).

Part 2:

Purpose

To use a spectroscope to observe the emission lines of elements.

Background

Each element has its own unique pattern of emission lines, like a fingerprint. The spectroscope is used to separate an element's emission lines, just as water droplets separate the wavelengths of white light to produce a rainbow.

Equipment

A spectroscope and an element lamp for helium and an element lamp for neon.

Procedure

1. You will observe the emission lines of helium and neon. For only helium, record the wavelength, in nm, for three lines in the visible region of light. Answer the questions about neon while you are observing the neon light.

2. Use the Rydberg equation to calculate each visible emission line wavelength (in nm units) for hydrogen. Do the calculated wavelengths agree or not agree with the known values? Show your calculations in your notebook worksheet.



Name:

RCBC CHE116 Notebook Worksheet for Experiment 11: Analysis of Drinking Water

Section 1

Experiment work was performed on	
Lab Partner:	

Section 2 Part 1: Raw Data

Part A: Preparing the Standard Solutions

Molarity of KMnO4 stock solution: _____

Size of volumetric flasks used: _____

Diluted to volume in flasks with: _____

Standard Solution Number	Concentration, M	Volume of Stock KMnO ₄ pipetted into flask, mL
1	1.0 x 10 ⁻⁴	
2	2.0 x 10 ⁻⁴	
3	3.0 x 10 ⁻⁴	
4	4.0 x 10 ⁻⁴	

Part B: Determining the best wavelength for absorbance

Wavelength, nm	Absorbance	Wavelength, nm	Absorbance
500		525	
510		530	
520		535	

Part C: Measuring the absorbance by the standard solutions and sample

Solution	Absorbance	Solution	Absorbance
Standard 1		Quality Control	
Standard 2		Unknown	
Standard 3			
Standard 4		The wavelengt analysis was	th used for this nm.

Part 2: Raw Data

Helium

Line color		
Observed		
Wavelength, nm		

Neon

Overall Color	
Color of the most intense emission lines	

Section 3: Experimental Procedure (Part 1 only)

Part A: The standard solutions for the calibration curve were prepared using the ______ M stock solution of potassium permanganate. The stock solution was pipetted into ______ mL volumetric flasks, and diluted to volume with DI water. For standard 1, ______ mL of stock solution was pipetted. For standard solution 2, ______ mL of stock solution was pipetted. For standard solution 3, ______ mL of stock solution was pipetted. For standard solution was pipetted.

Part B: The wavelength which had the best absorbance by the potassium permanganate was determined using standard solution _____. The absorbance of light at wavelengths 500 nm, 510 nm, 520 nm, 525 nm, 530 nm, and 535 nm was measured using the spectrophotometer. The zero was set at each wavelength with the blank. _____ was used as the blank.

Part C: The spectrophotometer was set at the λ_{max} determined in Part B as _____nm, and the absorbance of light by each standard solution, the quality control sample, and the unknown sample was measured. The spectrophotometer was zeroed before the absorbances were measured, using ______ as the blank.

Part D: The spreadsheet software ______ was used to create a calibration curve, using the standard solution concentrations and their absorbance values obtained in Part C. The concentration values were on the _____ axis and the absorbance values were on the _____ axis. The best-fit equation of the line was shown on the graph, along with the _____ value. The _____ sample and the _____ sample were not used to create the graph. The measured absorbance value of each sample solution was used, along the equation of the line, to calculate the concentration of KMnO₄ in each sample solution.

Section 4: Data Tables (Part 1 only)

Part B

Wavelength, nm	Absorbance	Wavelength, nm	Absorbance
500		525	
510		530	
520		535	

The wavelength at which the KMnO₄ absorbed the most light was _____ nm.

Part A and Part C

Standard Solution Number	Volume of stock solution pipetted, mL	Concentration, M	Absorbance at nm
1		1.0 x 10 ⁻⁴	
2		2.0 x 10 ⁻⁴	
3		3.0 x 10 ⁻⁴	
4		4.0 x 10 ⁻⁴	
QC sample	zero	To be calculated	
Unknown sample	zero	To be calculated	

Part D: See Calculations & Results

Section 5: Calculations & Results

Part 1Part A: Show the calculation for the amount to pipet to make standard
solution 1. You do not need to show the calculations for the other standard solutions.

Use Mc*Vc = Md*Vd

Part B: No calculations are needed.

Part C: **Attach the calibration curve graph** as the last page of this worksheet. Make sure the equation of the best-fit-line is printed on the graph, along with the R² value.

Part D: Show the calculation of the concentration of KMnO₄ in the quality control sample and the unknown sample. The absorbance value is the Y, calculate the concentration, which is the X, in the equation of the best-fit-line, Y = mX + b

Calculate the molarity of KMnO₄ in the quality control sample:

Calculate the molarity of KMnO₄ in the unknown sample:

Part 2

Helium (number of electrons in a helium atom:)					
Line color	Blue	Green / Blue	Yellow/Orange	Red	
Known Wavelength, nm	447	501	588	706	
Observed Wavelength, nm					

Neon (number of electrons in a neon atom:_____)

Overall light color	
Colors of most intense lines	

Hydrogen (number of electrons in a hydrogen atom:_____)

Line color	Blue	Green / Blue	Red
Known Wavelength, nm	434	486	656
Calculated Wavelength, nm			
Electron Transition $(5\rightarrow 2, 4\rightarrow 2, 3\rightarrow 2)$			

Show your calculations for each electron transition (use the Rydberg equation):

5 → 2	
4 → 2	
$3 \rightarrow 2$	

Section 6: Conclusion (Part 1 only)

The R² value and b value give some indication about how well the standard solutions were prepared. What was your R² value and b value and what does that mean about your standard solution preparation? Explain your answer.

The concentration of the quality control sample is known, and is listed in the Chemicals of the experiment document. Did your experimental result match up with this known value? What does this indicate about the accuracy of your analysis? Explain your answer.

The concentration of the unknown sample was calculated. Did the molarity result make sense, when you compare the unknown's absorbance value to the absorbance values of the standard solutions and their molarities? Explain your answer.

Experiment 12: Reaction Stoichiometry and the Formation of a Metal Ion Complex

(This experiment was adapted from Santa Monica College, Chemistry 11 and from https://chem.libretexts.org/Ancillary_Materials/Laboratory_Experiments/Wet_Lab_Experiments/General_C hemistry_Labs/Online_Chemistry_Lab_Manual/Chem_11_Experiments/08%3A_Reaction_Stoichiometry_ and_the_Formation_of_a_Metal_Ion_Complex_(Experiment).)

Purpose

To determine the reaction stoichiometry for the formation of a metal ion complex between iron(II) cations and 1,10-phenanthroline.

Background

The Method of Continuous Variation

Consider the study of a reaction where solutions of reactants A and B are mixed and product Q is formed:

$$x A + y B \rightarrow z Q$$

<u>The reactant mixtures are carefully chosen so that sum of the moles of A and B are constant</u>, and the amount of product that forms for each mixture is measured. This is known as the method of continuous variation. If either A or B is in excess, the excess will remain in solution rather than be used to form product. The maximum amount of Q is formed when A and B are mixed in the correct stoichiometric amounts, when there is just enough of each to react with nothing left over.

Suppose for example that x = 3 and y = 2, and that total number of moles of A and B is kept fixed at 0.10 moles. The amount of product formed will be at a maximum when the ratio A:B is 3:2, that is when A = 0.06 moles and B = 0.04 moles:



Note that to the left of the maximum on the plot (A<0.06 moles), there is not enough of A to react with all the B present. Thus, less than the maximum amount of product will be generated. To the right of the maximum (B<0.04 moles), there is not enough of B to react with all the A present. So once again, less than the maximum amount of product will be generated. As long as the total amount of A+B is constant, the maximum amount of product of product forms when the A:B ratio is the stoichiometric ratio for that reaction.

The reaction to be studied in this lab involves the formation of a metal ion complex. Metal ions, especially transition metal ions, possess the ability to form complexes with both organic and inorganic molecules called ligands. Here, iron(II) cations will be mixed with the ligand 1,10-phenanthroline to produce an iron(II)-phenanthroline complex:

m Fe²⁺ + n phen
$$\rightarrow$$
 Fe_m(phen)_n²⁺

Where phen is an abbreviation for 1,10-phenanthroline shown below:



Using the method of continuous variation as outlined earlier, several reactant solutions are prepared in which the mole quantities of the metal ion and the ligand are varied but the sum of the mole quantities is kept constant. The amount of complex produced will be measured, the maximum indicating when the correct stoichiometric ratio of Fe^{2+} :phen is used. (You will be determining the m and n in $Fe_m(phen)_n^{2+}$)

In this experiment, both the Fe^{2+} and phenanthroline solutions will have the same molarity concentration. Mixtures prepared with the same total number of moles will therefore have the same total volume (recall that moles = M x L). As an additional consequence, in each prepared mixture the volume ratio of reactants used will be identical to the mole ratio of reactants used. Therefore, it will be more convenient to analyze the amount of complex product formed as a function of reactant volumes, rather than as a function of reactant moles.

The spectrophotometer will be used to measure the amount of complex formed, since the complex is a red-orange color. You will need to determine the wavelength that has the maximum absorbance of light for this complex ion, then you will be able to do the stoichiometry part of the experiment. Since the iron(II)-phenanthroline complex is a redorange color, it is expected to absorb blue-green wavelengths, between 460 and 550 nm.

Chemicals

2.5 x 10^{-4} M Fe²⁺ solution (FeSO₄ and hydroxylamine hydrochloride) 2.5 x 10^{-4} M 1,10-phenanthroline solution Deionized water

Equipment

50-mL buret*, buret stand,	buret clamp
Plastic funnel	Spectrophotometer
Parafilm	Test tube rack

13 test tubes Beakers, small Plastic droppers

*Each group of 2 students gets one buret, either for the Fe²⁺ solution or the phenanthroline solution. Coordinate with the other group at your table.

Procedure

Part A: Determining λ_{max} for the Complex

1. Fill one test tube approximately 2/3 full with equal amounts of DI water and phenanthroline. This solution will be used as your calibration blank. Plastic droppers are fine for measuring the approximate volumes of each. Fill a second test tube approximately 2/3 full with a mixture of equal volumes of Fe⁺² and phenanthroline. Since this mixture will be used to find the λ_{max} , it does not have to be prepared with exact measurements. You can use plastic droppers to approximate the volumes of each solution used. Let this solution sit for approximately 10 minutes, to allow for color development.

2. Adjust the absorbance reading on the spectrophotometer to zero using the blank. Then measure the absorbance by the complex ion solution between 480 nm and 530 nm at 10 nm intervals. Note that each time the wavelength is changed the instrument must be recalibrated to zero with the blank. Record this data in your notebook worksheet and identify the wavelength of maximum absorbance of the complex (λ_{max}).

3. Save your blank for the next part of the experiment. Dispose of this complex ion mixture after you have identified λ_{max} .

Part B: Preparing Mixtures of Reactant Solutions

1. (One of the two groups) Obtain approximately 70 mL of Fe^{2+} stock solution. Rinse a buret with DI water. Then, rinse it three times, each with 2 mL of Fe^{2+} solution. Then add the remaining iron solution to your buret. Use your buret to add the Fe^{2+} solution to each of the 11 test tubes. The volume of the Fe^{2+} solution for each tube is:

Test Tube #	mL Fe ²⁺	Test Tube #	mL Fe ²⁺
1	4.50	7	1.50
2	4.00	8	1.00
3	3.50	9	0.75
4	3.00	10	0.50
5	2.50	11	0.25
6	2.00		

2. (One of the two groups) Obtain approximately 70 mL of phenanthroline solution. Rinse a buret with DI water. Then, rinse it three times, each with 2 mL of phenanthroline solution. Then add the remaining phenanthroline solution to your buret. Use your buret to add the phenanthroline solution to each of the 11 test tubes. The volume of the phenanthroline solution for each tube is:

Test Tube #	mL Phen	Test Tube #	mL Phen
1	0.50	7	3.50
2	1.00	8	4.00
3	1.50	9	4.25
4	2.00	10	4.50
5	2.50	11	4.75
6	3.00		

3. Mix each solution well by covering the test tube with Parafilm and inverting it several times. The red-orange color of the iron(II)-phenanthroline complex should be fully developed after ten minutes.

Do not let your iron(II)-phen solutions sit for more than 15 minutes.

4. Set the wavelength of the spectrophotometer to λ_{max} and again recalibrate it to zero using the blank. Since all remaining measurements will be taken at this wavelength, no further recalibration is required.

5. Measure the absorbance of each of the eleven prepared mixtures. Record this data in your notebook worksheet.

6. When completely finished, dispose of your waste in the designated waste bottle.

Part C: Data Analysis

1. Using Microsoft Excel or Google Sheets, graph your absorbance vs. volume of phenanthroline used. Treat ascending and descending values as two separate data sets, which will give you two separate graphs.

2. Add a best-fit trendline to each graph, and obtain the equations of these lines.

3. Solve for the optimum volumes, (the point of intersection of these lines) using your two equations, with the method of simultaneous equations.

4. From the point of intersection, you can determine the volume of reactants that yields the maximum absorbance (hence the maximum amount of complex formed).

Remember, since the concentrations of the reactant solutions are equal, the volume ratio is equal to the mole ratio.

5. Use this volume ratio to determine the simplest whole number ratio of Fe^{+2} :Phen, which is the stoichiometric ratio for this reaction.



Example Calculation:

Use the method of simultaneous equations to solve for x. That will give the volume of phenanthroline that will produce the maximum amount of product.

Experiment 12: Stoichiometry

Example Calculation

The total volume in each test tube was 5 mL.

 $Y = 0.2061 \text{ x} - 0.0119 \qquad Y = -0.5076 \text{ x} + 2.4938$ 0.2061 x - 0.0119 = -0.5076 x + 2.4938Now solve for x: 0.2061 x - 0.0119 = -0.5076 x + 2.4938 $0.7137 \text{ x} = 2.5057 \qquad x = (2.5057 / 0.7137) = 3.511$ $x = 3.511 \text{ mL} \qquad \text{optimum volume of phen} = 3.511 \text{ mL}$

optimum volume of $Fe^{2+} = 5.00 \text{ mL} - 3.51 \text{ mL} = 1.49 \text{ mL}$

The mole ratio (same as volume ratio) determined by this calculation is:

Fe ²⁺ 1.49 : Phen 3.51	now get whole-number subscripts	Fe^{2+} $\frac{1.49}{1.49}$: Phen $\frac{3.51}{1.49}$
Round off to whole numb	ers (3.51 / 1.49 = 2.36, round to 2)	
Fe ²⁺ 1 : Phen 2		

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Section 1

Experiment work was performed on _	
Lab Partner:	•

Section 2 Raw Data

Part A: Determining λ_{max} for the Complex

The solution color was allowed to develop for _____ minutes.

Wavelength, nm	Absorbance	Wavelength, nm	Absorbance
480		510	
490		520	
500		530	

Part B: Preparing Mixtures of Reactant Solutions

The solution color was allowed to develop for ______ minutes.

The molarity of the Fe²⁺ stock solution was ______ M.

The molarity of the phenanthroline stock solution was ______ M.

The λ_{max} used was _____ nm.

Test Tube #	mL Fe ²⁺	mL Phenanthroline	Absorbance at λ_{max}
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			

Section 3: Experimental Procedure

Part A: The wavelength which had the best absorbance by the complex ion was determined. The absorbance of light at wavelengths ______ nm through ______ nm was measured at ______ nm increments, using the spectrophotometer. The zero was set at each wavelength with the blank. A solution of ______ was used as the blank.

Part B: The spectrophotometer was set at the _____ nm λ_{max} determined in Part A, and the absorbance of light by each solution was measured. The spectrophotometer was zeroed before the absorbances were measured, using _____ as the blank.

Part C: The data obtained in part B was used to make two graphs. Each graph was made with absorbance on the _____-axis and mL of phenanthroline on the _____-axis. The volumes of iron and phenanthroline that gave the maximum amount of product were calculated using the equation of the best-fit lines. These volumes were used to determine the subscripts of the Fe-Phenanthroline complex ion.

Section 4: Data Tables

Part A

Wavelength, nm	Absorbance	Wavelength, nm	Absorbance
480		510	
490		520	
500		530	

Test Tube #	mL Fe ²⁺	mL Phenanthroline	Absorbance at nm
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			

Part B

Section 5: Calculations & Results

Part A:

The wavelength that had the highest absorbance value (λ_{max}) is: _____

Part B: Use the data in Part B to make the graphs for Part C.

Part C: The data obtained in Part B was used to graph absorbance (Y-axis) vs. mL phenanthroline (X-axis) for both the ascending and the descending data. **Attach the graphs as the last page of this worksheet.**

The best-fit equations of the lines and R² values are:

A P	1	D 2		
Acconding draph.	nact_tit lina	R4	_	
			_	

Descending graph: best-fit line	, F	२ 2	=	
---------------------------------	-----	------------	---	--

Calculate the optimum volume of phenanthroline (solve for X):
Calculate the optimum volume of Fe^{2+} (5.00 – X = opt. volume Fe^{2+}):
Use the optimum volumes as temporary subscripts for the complex ion:
Fe ²⁺ Phen
Calculate the whole-number subscripts for the iron-phenanthroline complex ion (see page 47 of the experiment document for the example calculation).
Fe ²⁺ Phen

Section 6: Conclusion

The R² value gives some indication about how well the solutions in Part B were prepared. Were the data points for these solutions all on the best-fit-lines? For a complex ion, there is one metal ion with one or more things attached. Did your ratio of Fe and Phenanthroline work out that way; 1 Fe with 1 or more phenanthroline? Explain your answers.

Experiment 13: Molecular Weight of Carbon Dioxide

(This experiment is from Santa Monica College, Chemistry 11)

Purpose

The purpose of this experiment is to determine the molecular weight of carbon dioxide, using the ideal gas law. The experimental molecular weight determined will be compared to the known molecular weight of carbon dioxide.

Background

The molecular weight (MW) of a substance is determined by:

The grams can be determined by using the lab balance. The moles can be calculated with the ideal gas law, PV = nRT.

Therefore, in this experiment, you will measure the **mass** of the CO₂ sample, as well as the **temperature**, **pressure**, and **volume** of the gas sample. You will calculate the **moles** of the gas sample

The carbon dioxide gas is produced by the reaction of calcium carbonate and hydrochloric acid:

 $CaCO_{3}\left(s\right) \ + \ 2 \ HCI_{(aq)} \ \rightarrow \ CO_{2}\left(g\right) \ + \ CaCI_{2}\left(aq\right) \ + \ H_{2}O\left(I\right)$

The carbon dioxide gas will be collected in an Erlenmeyer flask that is covered with aluminum foil. The aluminum foil and the fact that carbon dioxide gas is denser than air slows the rate at which the carbon dioxide mixes with the air outside of the flask.

The **mass** of the gas will be determined by mass-by-difference. Be sure to obtain the mass of the empty flask covered by the foils, and then the mass of the foil covered flask filled with carbon dioxide. Before subtracting these two masses to get the mass of the CO₂, the mass of the air in the 'empty' flask must be accounted for.

The **temperature** of the gas will be measured by briefly placing a thermometer underneath the foil, after the final mass of the gas sample is obtained.

The **volume** of the gas will be equal to the volume of the flask. The volume of the flask will be measured by filling it to the brim with tap water, and measuring that water's volume with a graduated cylinder. This is done after the mass and temperature are determined.

The **pressure** of the gas will be equal to the pressure in the room, since the flask is not sealed. The pressure in the room can be read from the barometer.

The mass of the air will be calculated with the volume of the flask and the density of air. See Table 1 for the density of dry air. Assume the air in the lab is dry.

Density = (mass / volume)	mass = (Density · volume)
Table 1. Density of Dry Air	

		Density, g/L	
Temperature, C	P = 750 torr	P = 760 torr	P = 770 torr
17	1.201	1.217	1.233
18	1.197	1.213	1.229
19	1.193	1.209	1.225
20	1.189	1.205	1.221
21	1.185	1.201	1.216
22	1.181	1.197	1.212
23	1.177	1.193	1.208
24	1.173	1.189	1.204
25	1.169	1.185	1.200

Information from the CRC Handbook of Chemistry and Physics, 64th ed., 1983-4

Calculate the mass of the dry air, and subtract that value from the mass of the 'empty' flask. This will give the true mass of the 'empty' flask.

mass of $CO_2 = (mass CO_2 + flask + foils) - (mass flask + foils + air) - (mass air))$

Chemicals

Al foil, approximately 15 cm x 15 cm, 2 pieces (one with a hole and one without a hole) CaCO₃, in the form of marble chips (limestone)

CaCl₂, anhydrous solid, for the drying tube

HCI, 6 M (HCI can cause serious chemical burns and blindness) Water, deionized and tap

Equipment

One experimental setup (Figure 1) Figure 1: Gas Generation Setup at RCBC



1. Obtain one 250 mL Erlenmeyer flask. This will be designated as flask **B**. Make sure that the flask is both clean and dry. Record the mass of the flask and both pieces of foil to the nearest 0.001 g using the balance. Note that it is mass (flask + foils + air) that is being measured here. Also record the temperature of the air in the flask. Keep these two pieces of foil. If you need to get a new piece of foil, you have to repeat the step.

2. Place about 25 grams of marble chips into the jar designated as flask **A**. Add enough deionized water to cover the chips. Insert the two-holed rubber stopper (with the thistle tube and the bent tube) into flask *A*, making sure that the thistle tube is adjusted so that it is beneath the water but not touching the bottom of flask. The stopper must fit tightly into the flask.

Also obtain about 25 mL of hydrochloric acid (6 M HCl), in a small, labeled beaker.

3. Assemble the apparatus shown in **Figure 1**. Insert the straight glass tube into flask B by placing it through a premade starter hole in one of the foil pieces. Attach the flexible end of the tubing to the drying tube. The flexible tubing attached to the bent glass tubing should be inserted into the other end of the drying tube.

4. When all is ready slowly pour approximately 10 mL of the hydrochloric acid into the top of the thistle tube and allow it to run through the tube and into flask **A**. The reaction should begin immediately as evidenced by gas evolution. Allow the reaction to continue for at least 20 minutes to displace all of the air in flask **B** with carbon dioxide gas. <u>During this time pay attention to what is happening in flask **A**; if the gas evolution ceases, add additional HCI solution through the thistle tube. (Don't let solution rise up into the stem of the thistle tube; raise the tube as needed.) After the 20 minutes remove the tube from flask **B** (keep the foil in place) and **immediately cover flask B with the second piece of aluminum foil.** Then weigh the flask containing carbon dioxide on the analytical balance to the nearest 0.001 g.</u>

5. Reassemble the apparatus and allow gas evolution to continue and flow into flask **B** for an additional 15 minutes. Again, weigh flask **B** (with both pieces of foil). Next, measure and record the temperature of the carbon dioxide in the flask. Use the barometer to read the atmospheric pressure.

6. Finally, in order to determine the volume of flask **B**, fill this flask with <u>tap water</u> to the brim. Use a larger graduated cylinder to measure the volume of water in the flask. Since the flask does not have a pouring lip, pour the water from the flask into a beaker, and then from the beaker into the graduated cylinder.

Calculations / Results section: Show all of your calculations in your notebook worksheet.

- 1. Convert all data units to the correct units for calculations.
- 2. Calculate the mass of the air in the 'empty' flask massair = (density · volume)
- 3. Calculate: mass of CO₂ = (mass cO₂ + flask + foils) ((mass flask + foils + air) (mass air))
- 4. Calculate the moles of CO_2 , using PV = nRT (use correct units)
- 5. Calculate the molecular weight of CO_2 . MW = grams CO_2 / moles CO_2
- 6. Calculate the %error, and comment on the %error in your conclusion.
 %Error = ((true MW experimental MW) / true MW) x 100

Name:

RCBC CHE116 Notebook Worksheet for Experiment 13: MW of CO₂ Since this experiment is short and is the last experiment before the lab practical, this notebook worksheet is due by the end of today's lab class.

Section 1

Experiment work was performed on _	
Lab Partner:	

Section 2 Raw Data

Temperature of the air in the room (which will be the same as in the fla	sk) =°C
Mass of 250 mL Erlenmeyer flask + 2 Al foils + air =	_ g
Molarity of the HCl acid used =	
Amount of time that the reaction happened, 1 st time =	_minutes
Mass of 250 mL Erlenmeyer flask + 2 Al Foils + CO ₂ =	g
Amount of time that the reaction happened, 2 nd time =	minutes
Mass of 250 mL Erlenmeyer flask + 2 Al Foils + CO ₂ =	g
Temperature of the CO ₂ in the flask = °C	
Atmospheric pressure, measured with barometer =	_ cm Hg
Total measured volume of the Erlenmeyer flask =	mL
Density of the dry air (obtain from Table 1) = $\ g / L$	

Section 3: Experimental Procedure

The equipment shown on a page 2 of the experiment document, and better shown in the lab room, was set up. The mass of flask B with the two pieces of Al foil + air was measured before the reaction occurred. Approximately ______ g of marble chips were put into jar A. Approximately ______ mL of the ______ M HCl solution was poured over the marble chips to cause the reaction between CaCO₃ and HCl to occur. The CO₂ gas flowed from flask A, through the drying tube of CaCl₂, and into flask B. The reaction was allowed to occur for ______ minutes, adding more HCl to flask A as needed to prevent the reaction from stopping. The mass of flask B with the two pieces of Al foil + CO₂ was measured after the reaction occurred for approximately 20 minutes. This was repeated after the equipment was reassembled and the reaction allowed to occur again for approximately ______ minutes. The final mass of flask B with two pieces of Al foil + CO₂ was measured.

Section 4: Data Tables

Parameter being measured	Value in units used in the lab	Value in units needed for PV=nRT
Flask B + 2 foils + air (before reaction)	g	
Flask B + 2 foils + CO ₂ (after first 20 minutes)	g	Use the larger of
Flask B + 2 foils + CO ₂ (after additional 15 minutes)	g	your calculations.
Temperature of CO ₂ in flask B	°C	К
Atmospheric pressure in the lab room	cm Hg	atm
Volume of flask B	mL	L

Section 5: Calculations & Results

Show the calculation of the temperature of CO₂ in units of Kelvin.

Show the calculation of the atmospheric pressure in units of atmospheres.

Show the calculation of the flask's volume in units of Liters.

(continued on the next page)

Mass of dry air in the 'empty' Erlenmeyer flask = (density x volume) (see Table 1)

Mass of $CO_2 = (mass flask+2 foils+CO_2) - [(mass flask+2 foils+air) - (mass dry air)]$

Moles CO_2 ; calculate with PV = nRT

MW CO_2 = mass of CO_2 / moles CO_2

% Error

Section 6: Conclusion

The true molecular weight of carbon dioxide is known. How did your experimental result compare to the true, known molecular weight? This is what the % error indicates. A % error less than 5% indicates that your experimental result is accurate. Were there any known sources of error?

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Review Questions for the Lab Practical Exam

(This is only a partial review. The answers are on the following page.)

- 1) The equation of a best-fit-line is given as Y = 87.6 X + 0.0003The absorbance of an unknown solution is measured as 0.682 What is the concentration of the unknown solution?
- 2) Use the Solubility Guidelines tables to answer these questions:

Will ZnCO₃ dissolve when put in water? (Is it (aq) or (s) ?)

What ions are present in solution when (NH₄)₃PO₄ dissolves in water?

What is the precipitate that forms when $Cu(NO_3)_2$ and $(NH_4)_3PO_4$ are mixed together?

Write a balanced reaction for when $Cu(NO_3)_2$ and $(NH_4)_3PO_4$ are mixed together.

3) Use the Activity Series table to answer these questions:

Cr(s) is put into a solution of acid, H¹⁺. A reaction takes place.
Is chromium oxidized or reduced? Is hydrogen oxidized or reduced?
Write the half-reaction for chromium. Write the half-reaction for hydrogen.
Write the whole redox reaction; make sure it is balanced.

4) Given the information in the table below, calculate the density of the metal.

Mass of the metal	2.684 g
Volume of water in the cylinder	5.00 mL
Volume of water + metal in the cylinder	6.71 mL
Volume of only the metal	
Density of the metal	

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5) Miscellaneous questions:

What is the concentration of CuCl₂ when 3.0 mL of a 1.0×10^{-2} M solution is diluted to 50.0 mL?

How much CuCl₂ stock solution do you need to make 100.0 mL of a $5.0x10^{-3}$ M solution? The stock solution is $1.0x10^{-1}$ M.

If you had 4.683 g of CuCl₂, how many moles of CuCl₂ would you have?

If you had 0.082 moles of CuCl₂, how many grams of CuCl₂ would you have?

If you had 0.0780 moles of CuCl₂, how many moles of Cl¹⁻ would you have?

Answers:

- 1) 0.00778
- 2) No, $(3 \text{ NH4}^{1+} + 1 \text{ PO4}^{3-})$, $Cu_3(PO_4)_{2(s)}$, $3Cu(NO_3)_{2(aq)} + 2(NH_4)_3PO_{4(aq)} \rightarrow Cu_3(PO_4)_{2(s)} + 6 \text{ NH4}NO_{3(aq)}$
- 3) Chromium is oxidized, Hydrogen is reduced $Cr_{(s)} \rightarrow Cr^{3+}_{(aq)} + 3e - 2H^{1+}_{(aq)} + 2e - \rightarrow H_{2(g)}$ $2Cr_{(s)} + 6 H^{1+}_{(aq)} \rightarrow 2 Cr^{3+}_{(aq)} + 3H_{2(g)}$
- 4) Volume of only the metal is 1.71 mL, density of the metal is 1.57 g/mL
- 5) 0.00060 M, 5.0 mL, 0.03482 mole, 11 g, 0.156 mole Cl¹⁻