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 Δ 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, $4.184 \text{ J} \cdot (^{\circ}C^{-1} \cdot g^{-1})$,

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 $(ΔH > 0)$.

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:

 $\Delta H_{rxn} = (-1) \cdot ((m \cdot C \cdot \Delta T) + (C_{Cal} \cdot \Delta T))$ where C_{Cal} is the calorimeter constant and it is calculated to be $C_{\text{Cal}} = 10.0 \text{ J} \cdot \text{°} \text{ C}^{-1}$ 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): HNO₃(aq) + NaOH (aq) \rightarrow NaNO₃(aq) + H₂O (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)*

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

Plugging all the values:

mass of solution = $(1.00g \cdot mL^{-1} \cdot 12.00mL) = 12.0g$ ΔHrxn =(-1) **(** (12.0 g · 4.184 J ·◦ C−1 ·g−1 ·9.0◦C−1) + (10.J ·◦ C−1 ·9.0◦C) **)**

Solving for ΔH_{rxn} we have −542 J. The number of moles of HNO₃ is: n =0.01200 L \times 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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HCl + NaOH \rightarrow NaCl + H_2O \Delta H_1and
NH_3 + HCl \rightarrow NH<sub>4</sub>Cl \Delta H_2or
NaOH + NH<sub>4</sub>Cl \rightarrow NaCl + NH<sub>3</sub> + H<sub>2</sub>O \Delta H<sub>rxn</sub>
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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:

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

Chemicals

 $HCI_{(aq)}$, $NH_{3(aq)}$, NaOH $_{(aq)}$, NH₄C $I_{(aq)}$ (Caution: All solutions are 1.0 M concentration.)

(Note: When NH₃ is in solution, it is typically written as $NH₄OH_(aa)$. 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 THE SAME 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 ΔH for the HCl, 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.
- 7) 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 = $(d \cdot V)$ 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 HCl, 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.
- 4) Measure the temperature of each of these solutions. 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.
- 7) 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 = $(d \cdot V)$ where V is the volume of $(\text{acid} + \text{base})$ used. $\Delta H_2 = (-1)$ ((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).

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.
- 4) Measure the temperature of each of these solutions. 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 to gently mix the solutions.
- 7) 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 = $(d \cdot V)$ where V is the volume of (acid + base) used. $\Delta H_{rxn} = (-1)$ ((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).

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 for each trial were: acid,base________________________, volume of <u>acid</u> used per trial: __________ acid,base__________________________, volume of <u>acid</u> used per trial: __________

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

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

Acid, Base Combination: NH4Cl, NaOH

Chemical reactions used:

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

Show your calculated results here:

Acid, Base Combination: HCl, NH³

Hess's Law

Calculation ΔH_{rxn} for NH₄Cl + NaOH \rightarrow NaCl + NH₃ + H₂O

Using your calculated results for:

HCl + NaOH → NaCl + H2O ΔH1average = _________

 $HCl + NH_3 \rightarrow NH_4Cl$ $\Delta H_{2average} =$ $\frac{1}{2}$

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 ΔH1average result agree with the expected -52 kJ/mole? (See the bottom of page 3 in the experiment document.) Did your calculated Hess's Law ΔHrxn result agree with your experimental ΔHrxn 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 acidbase reaction finishes. For example, in the reaction between acetic acid (CH3COOH) and sodium hydroxide (NaOH):

 $CH₃COOH + NaOH \rightarrow CH₃COONa + H₂O$

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 H^{1+} 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) Pipet, 10 mL, and pipettor Erlenmeyer flask, 125 mL Funnel Volumetric flask, 100 mL

Plastic droppers 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.
- 3) 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.)

- 1) 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. *Record all buret volumes with two digits after the decimal point.*
- 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

- 1) 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.
- 2) Pipet 20.0 mL of the **diluted** 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. *Record all buret volumes with two digits after the decimal point.*
- 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 HCl 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.

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

deviation $1 = / \text{trial} 1$ - average / deviation $2 = / \text{trial} 2$ - average / deviation $3 = /$ trial $3 -$ average /

Section 3: Experimental Procedure

A _______mL buret was cleaned and filled with _______M NaOH solution. _______mL of the HCI 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. The 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.

Section 4: Data Tables

Section 5: Calculations & Results

For the titration of HCl

Moles NaOH used, Trial 1:

Molarity HCl, Trial 1:

Average Molarity HCl:

Standard Deviation:

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

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:

For the commercial vinegar calculations:

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

Section 6: Conclusion

The molarity of HCl 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 $[H¹⁺]$ or $[H₃O¹⁺]$). 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 $[H¹⁺] = 1.0 \times 10^{-5}$ 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

Equipment

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 DI 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 ________________________ Lab Partner: __

Section 2 Raw Data

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

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.

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

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 The the same 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.

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.)

Parts 3 and 5: For only the HCl solution:

Calculated concentration of H1+ from the measured pH values.

Calculate the $[H¹⁺]$ with the measured pH values as indicated.

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.

The concentration of H^{1+} equals the concentration of HCl because there is one H¹⁺ for every one HCl, and HCl is a strong acid (100% ionized).

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

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-potassiumpermanganate/). 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 Beakers, assorted small sizes Test tubes, small 13x100 size Pipet, 10 mL, and pipettor Parafilm Plastic droppers Test tube rack 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.

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

- 1) Obtain enough concentrated potassium permanganate solution from the stock bottle in the hood $(5.0 \times 10^{-3} \text{ M})$. 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.
- 4) 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

- 1) 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 R^2 value of 1. The Y-intercept, b, should be zero.)
- 2) Use the standard solution concentrations as the x axis values. Their absorbance values go on the y axis. **DO NOT GRAPH** 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

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: _____________________

Part B: Determining the best wavelength for absorbance

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

Part 2: Raw Data

Helium

Neon

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 4, ________ mL of stock 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. **Example 2** 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 example a 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 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

The wavelength at which the $KMnO_4$ absorbed the most light was mm .

Part A and Part C

Part D: See Calculations & Results

Section 5: Calculations & Results

Part 1 Part 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:

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

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

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

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 R^2 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:

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

The reactant mixtures are carefully chosen so that sum of the moles of A and B are constant, 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 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
$$
\rightarrow
$$
 Fem(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 Fe²⁺:phen is used. (You will be determining the m and n in Fe_m(phen)_n²⁺)

In this experiment, both the $Fe²⁺$ 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 $= M \times L$). 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

*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 λmax 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 λmax.

Part B: Preparing Mixtures of Reactant Solutions

1. (One of the two groups) Obtain approximately 70 mL of Fe2+ stock solution. Rinse a buret with DI water. Then, rinse it three times, each with 2 mL of Fe²⁺ solution. Then add the remaining iron solution to your buret. Use your buret to add the $Fe²⁺$ solution to each of the 11 test tubes. The volume of the $Fe²⁺$ solution for each tube is:

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:

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.

Example Calculation

The total volume in each test tube was 5 mL.

 $Y = 0.2061 x - 0.0119$ $Y = -0.5076 x + 2.4938$ 0.2061 x – 0.0119 = -0.5076 x + 2.4938 Now solve for x: $0.2061 x - 0.0119 = -0.5076 x + 2.4938$ 0.7137×2.5057 $x = (2.5057 / 0.7137) = 3.511$ $x = 3.511$ mL optimum volume of phen = 3.511 mL

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

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

 $Fe²⁺$ 1.49 : Phen 3.51 now get whole-number subscripts Fe²⁺ $\frac{1.49}{1.49}$: Phen $\frac{3.51}{1.49}$ Round off to whole numbers $(3.51 / 1.49 = 2.36$, round to 2) $Fe²⁺$ ₁ : Phen ₂

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RCBC CHE116 Notebook Worksheet for Experiment 12: Reaction Stoichiometry Name: ___

Section 1

Section 2 Raw Data

Part A: Determining λ_{max} for the Complex

The solution color was allowed to develop for ____________ minutes.

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 λmax used was ____________ nm.

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 $\sum_{m=1}^{\infty}$ 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

Part B

Section 5: Calculations & Results

Part A:

The wavelength that had the highest absorbance value (λ_{max}) is: $\frac{1}{\lambda_{max}}$

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:

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:

$$
MW = \frac{grams}{moles}
$$

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:

 $CaCO_{3 (s)} + 2 HCl_(aq) \rightarrow CO_{2 (g)} + CaCl_{2 (aq)} + H₂O_(l)$

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.

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 $CO_2 = (mass \cos \theta + \text{f} \sin \theta) - (mass \sin \theta + \text{f} \sin \theta + \text{g} \sin \theta) - (mass \sin \theta)$

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

HCl, 6 M *(HCl 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 $(flass + foils + air)$ 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. During this time pay attention to what is happening in flask **A**; if the gas evolution ceases, add additional HCl 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.

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 tap water 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 $CO_2 = (mass \cos \theta + \text{flask} + \text{foils}) ((mass \text{flask} + \text{foils} + \text{air}) (mass \text{air}))$
- 4. Calculate the moles of $CO₂$, using $PV = nRT$ (use correct units)
- 5. Calculate the molecular weight of $CO₂$. MW = grams $CO₂$ / moles $CO₂$
- 6. Calculate the %error, and comment on the %error in your conclusion. %Error = ((true MW – experimental MW) / true MW) \times 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

Section 2 Raw Data

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 example of the 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

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 CO_2 = (mass flask+2 foils+ CO_2) – $[$ (mass flask+2 foils+air) – (mass dry air) $]$

Moles $CO₂$; calculate with $PV = nRT$

MW $CO₂$ = mass of $CO₂$ / moles $CO₂$

% 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 $Y = 87.6 X + 0.0003$ The 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 $Cu(NO₃)₂$ and $(NH₄)₃PO₄$ are mixed together?

Write a balanced reaction for when $Cu(NO₃)₂$ and $(NH₄)₃PO₄$ are mixed together.

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

 $Cr(s)$ is put into a solution of acid, H^{1+} . 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.

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5) Miscellaneous questions:

What is the concentration of CuCl₂ when 3.0 mL of a $1.0x10^{-2}$ 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, $(3 NH_4^{1+} + 1 PO_4^{3-})$, Cu₃ $(PO_4)_{2(s)}$, $3Cu(NO_3)_{2(aq)} + 2(NH_4)_{3}PO_{4(aq)} \rightarrow Cu_3(PO_4)_{2(s)} + 6NH_4NO_{3(aq)}$
- 3) Chromium is oxidized, Hydrogen is reduced $Cr_{(s)} \rightarrow Cr^{3+}(aq) + 3e- 2H^{1+}(aq) + 2e- 3H^{2}(q)$ $2Cr(s) + 6 H^{1+}(aq) \rightarrow 2 Cr^{3+}(aq) + 3H_{2}(q)$
- 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⁻