

Faculty of Engineering
Chemical Engineering and Applied Chemistry



General Chemistry

Laboratory Manual



ATILIM UNIVERSITY

ANKARA

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GENERAL CHEMISTRY LABORATORY RULES

1. You are responsible for all safety rules in the lab manual.
2. You have to wear a **LAB COAT** and **safety goggles** and also have a **lab manual** and **calculator** while performing experiments in the laboratory. Without them you are not allowed to enter the lab section. Safety goggles will be given by your assistants.
3. Read the lab experiments and any suggested additional reading(s), before coming to lab.
4. **You will take a quiz at the beginning of the experiment.**
5. **Eating, drinking, smoking, and cell phones are forbidden** in the laboratory at all times. Avoid unnecessary movement and talk in the laboratory.
6. Any accident involving even the most minor injury must be reported to the lab assistants.
7. Do not attempt any unauthorized experiment. Perform only lab operations and activities.
8. **Cheating** will lead to a **zero point** for quizzes and lab reports. If it is repeated second time, you will fail from the course and get a **disciplinary warning**.
9. You have to check your data sheet with your assistants at the end of the lab period.
10. **If you miss more than one of the experiments without an excuse, then you will fail from the lab and also fail from CEAC 105 course.**
11. In order to **pass the lab** you have to get **50 points** over 100 points at the end of the semester in the laboratory (Quizzes: 20%, Lab Reports: 50%, Opinion: 30%).
12. To be able to take a make up from any laboratory experiments, you should bring an approved a medical report to the General Chemistry Group Coordinator.
13. If **you fail from the lab**, you will also **fail from the course**.

LABORATORY EQUIPMENT

		
<p>Beakers</p>	<p>Erlenmeyer Flask</p>	<p>Graduated Cylinder</p>
		
<p>Pisette</p>	<p>Thermometer</p>	<p>Burette</p>
		

<p>Test Tube</p>	<p>Condenser</p>	<p>Volumetric Flask</p>
		
<p>Funnel</p>	<p>Test Tube Rack</p>	<p>Test Tube Holder</p>
		
<p>Curicible</p>	<p>Weighing Boat</p>	<p>Balance</p>
		
<p>Clamp</p>	<p>Lab Stand</p>	<p>Bunsen Burner</p>



Hood



Goggles



Glove

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Experiment 1
Introduction to Laboratory Techniques



Purpose: To reinforce the understanding of some common laboratory concepts and techniques while gaining knowledge in data treatment by reporting. In the first part of the experiment, potassium permanganate solution (solid KMnO_4 dissolved in water) will be separated into its constituents by distillation, and in the second part, determination of the solubility of a pure substance in a given liquid, or, in the case of two liquids, and the miscibility tests will be done. To reinforce the understanding of some common laboratory concepts and techniques while gaining knowledge in data treatment by reporting. In the first part of the experiment, potassium permanganate solution (solid KMnO_4 dissolved in water) will be separated into its constituents by distillation, and in the second part, determination of the solubility of a pure substance in a given liquid, or, in the case of two liquids, and the miscibility tests will be done.

Pre-laboratory Work

Before the experiment in the laboratory, you should be able to answer these questions.

- 1) List five physical properties.
- 2) What are the differences between physical and chemical changes?
- 3) Define solubility and discuss the factors affecting solubility.
- 4) What is density? How do you calculate it?

Theory

Separation of Substances

All material things which have mass and occupy space in universe referred to as **matter**. Every substance has a large number of physical and chemical properties. **Physical properties** are the characteristics of a substance that can be seen without changing the composition of it. Common physical properties include color, smell, taste, solubility, density, electrical conductivity, heat conductivity, melting and boiling points. When a physical change is observed, the substance retains its chemical identity, but loses only its appearance. For example, when ice is melted, only a change of the state occurs, no new substance is formed.

On the other hand, **chemical properties** represent the changes in the composition of a substance when it reacts with other substances or decomposing into new other pure substances. Chemical properties include decomposition by heating, and reactions of the substance with water, oxygen, acids, bases. When chemical changes are observed, new substances are formed that have totally different properties and compositions considering to starting material. For example, when methane, the main component of natural gas, burns by reacting with oxygen in the air, carbon dioxide and water are formed as the new products.

In other words, while physical changes are **reversible**, chemical changes are **irreversible** (not reversible).

Solutions

When a solid is mixed with a liquid and dissolves in that liquid, the resultant mixture formed is called a **solution**. This liquid solution may contain no visible solid particles and it may be colorless or have a characteristic color. Solutions are **homogenous mixtures**. When a solution forms, it can be stated that "The solid dissolves in the liquid" or "The solid is soluble in the liquid". The constituents of a solution are **solute** (minor fraction), and **solvent** (major fraction)

Solubility and Miscibility

Solubility can be defined as the amount of a particular substance that can dissolve in a particular solvent. The maximum amount of a particular substance that can be dissolved in 100 mL of pure water at a particular reference temperature is known as percent solubility. When a solid dissolves in a liquid, it is said to be **soluble** in that liquid. If the solid does not dissolve, then it is **insoluble**.

Miscibility is taken into account when two liquids are mixed. If this mixture is completely uniform in appearance, in this case the liquids are said to be **miscible**. If individual layers are formed when they are poured together, then these two liquids are not miscible at all, i.e. they are **immiscible**.

Density

Density is simply defined as “mass per unit volume”. If you wonder how dense a material is, you have to know the mass and volume of it. Then, you can easily find the density of the substance by using this formula: $\rho = m / V$

The unit of the density is g / mL or g / cm³.

In Part A, water will be separated from potassium permanganate by *distillation*. When the potassium permanganate solution is heated, water vapor will be driven off first, because the boiling point of potassium permanganate is much higher than that of water.





In Part B, The behavior of two substances on a solvent will be tested. For these two chemicals, solubility in water will be examined. For the dissolving one, solubility amount will be found. Then, miscibility of some liquids will be tested.

In Part C, Density of water will be measured in two different ways. Then, the density of an unknown solid will be calculated.

Materials

Potassium permanganate (KMnO ₄)	Diethyl ether	50 mL beakers
Starch	Ethyl alcohol	Balance
Sodium carbonate (Na ₂ CO ₃)	Bunsen burner	Graduated cylinder
Distilled water	One holed rubber stopper	Ring, clamp
Any solid particle	Boiling chips	

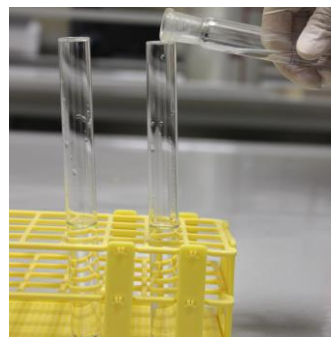
Procedure

Part A: Separation by Distillation	
<p>1. Pour 30 mL potassium permanganate solution (solid KMnO_4 dissolved in water) into a 100 mL round bottom flask. Add some boiling chips into the flask to make solution boil calmly.</p>	
<p>2. Set a simple distillation apparatus by inserting the short end of the glass tubing acting as condenser in a one-holed rubber stopper.</p>	
<p>3. Ask your assistant how to use the Bunsen burner. Light it and adjust until you have a small and continuous hot flame.</p>	
<p>4. Heat the KMnO_4 solution and observe the hot solvent vapors of the solution are cooled and dripped into the test tube. Continue distillation process until about 10 mL of liquid have distilled over. Observe the differences in color between the distillate and the original solution; write these observations on your data sheet.</p>	

Part B: Identifying Substances by Their Properties

B.1. Solubility in Water

1. Take clean two test tubes and fill them with 20 ml of water.

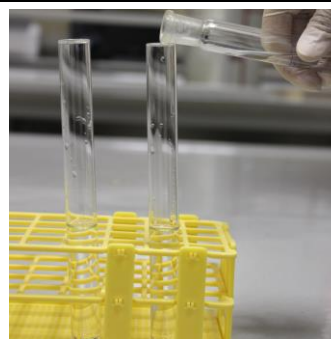


2. Take small amount (approximately half of a pea) of sodium carbonate; Na_2CO_3 , and starch and place them into test tubes that contain water. Shake the tubes gently and observe whether or not the substances dissolve. Some substances dissolve slowly, in this case set the test tube aside for few minutes and examine again. Are the compounds soluble or insoluble? Record your observations.



B.2. Miscibility

1. Take clean two test tubes and fill half of them with water.



2. Now, to the first tube, put some amount of alcohol and to the second one, put diethyl ether. Shake the test tubes gently or mix the contents with stirring rod. Observe what happened. Which one is miscible, record your observations on your data sheet.



Part C: Density Measurement

C.1. Density of Water

1. Now take a clean and dry graduate cylinder and weigh it. Then put 20 mL water in it and weigh again to find the mass of water. Carry out the density calculation and find the density of water.



C.2. Density of a Solid

1. Take an irregular shape solid from your assistant and weigh it.



2. To find volume of the solid, put it into graduated cylinder that contains 20 ml water in the previous part and calculate the volume of the solid according to increase in the water level. Carry out the density calculation to find the density of the solid material.



DATA SHEET

Introductory to Laboratory Techniques

Student's Name :

Date:

Laboratory Section/Group No :

Assistant's Name and Signature:

A. Separation by Distillation:

- i. What was the color of the original (KMnO_4) solution?

- ii. What is the color of the distillate which is collected in the test tube?

- iii. Why did we collect water as distillate in test tube? (Hint: Consider the boiling point differences of distinct substances)

B. Identifying Substances by their Properties

	Solubility in water (soluble or insoluble)
Starch	
Na_2CO_3	

	Miscibility (miscible or immiscible)
Water - Alcohol	
Water- Diethyl ether	

C. Density

- i. Density of water while you use graduated cylinder:

- ii. Density of the irregular shape solid:

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Experiment 2
The Law of Definite Proportions



Purpose: To understand “ the law of definite proportions” concept and to learn how to make gravimetric analysis (analysis by weighing) calculations.

Prelaboratory Work

Before the experiment in the laboratory, you should be able to answer these questions.

1. Define the terms "compound" and "catalyst".
2. How do empirical and molecular formulas differ?
3. Calculate the percent composition of HNO_3 ? (H: 1 g/mol; N: 14 g/mol; O: 16 g/mol)
4. What is law of definite proportions?
5. How many lithium atoms are present in 0.01456 g of lithium?
6. What is percent oxygen by weight in water molecule?
7. Given that zinc chloride has a formula weight of 136.28 g/mol, what is its formula?

Theory

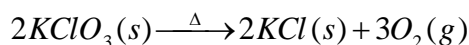
The law of definite proportions states that a chemical compound always contains exactly the same **proportion** of elements by mass. Law of definite proportions shows a good way to find percent weight or exact weight of a desired element in a compound. It also gives useful information to find empirical or molecular formula for a compound and percent weight of a compound in an unknown mixture.

Example: What is the % O by weight in V_2O_5 ? (Atomic weights are; V: 50.9 g/mol, O: 16 g/mol)

Solution: First, we must find the total weight of the compound. Then we will divide the desired element's weight by the total weight as in follow:

$$O\% = \frac{5 \times (16.0)}{2 \times (50.9) + 5 \times (16.0)} \times 100 = 44.0\%$$

In today's experiment, potassium chlorate ($KClO_3$) will be decomposed into potassium chloride (KCl) and oxygen (O_2) by heating (MnO_2 will be used as a catalyst to speed up the reaction without being consumed.) :



As it is seen from the reaction equation, oxygen gas releasing upon decomposition results with the weight loss of initial compound. In other words, the weight difference gives the weight of oxygen in the compound.

At the end of the experimental part, theoretical and experimental percent oxygen by weight for $KClO_3$ will be calculated and compared. Comparison will give the Percent Error for the experiment.

Percent error is the ratio of the absolute value of the error to the theoretical value and multiplied by 100.

Error: experimental value – theoretical value

$$\% \text{ Error: } \frac{|\text{experimental} - \text{theoretical}|}{\text{theoretical}} \times 100$$

Example: A chemical compound theoretically contains 39.2 % O by weight. In a laboratory, % O by weight for this compound was found as 36.3 %. Calculate the error and % error of this experiment.

Solution:

$$\text{Error} = 36.3 - 39.2 = -2.9$$

$$\% \text{ Error} = \frac{|36.3 - 39.2|}{39.2} \times 100 = 7.4\%$$

Materials

Manganese (IV) oxide (MnO ₂)	Test tube	Bunsen Burner
Potassium chlorate (KClO ₃)	Balance	Wooden Tongs
Potassium chloride (KCl)		

Procedure

Part A: Percent Oxygen in Potassium Chlorate

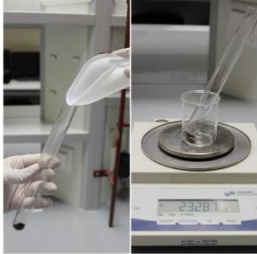

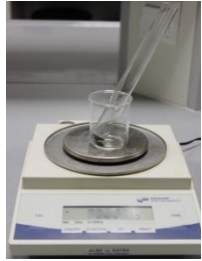

Drying the Catalyst

1. Put about a tea-spoon of MnO_2 in a dry test tube. Heat the test tube in order to remove the moisture of the catalyst. Move the test tube continuously on the flame.



2. After test tube is cooled to room temperature, weigh it (W_1).



Decomposition Reaction	
<p>1. Add about between 2 - 4 g of KClO_3 into the test tube and weigh again (W_2). Calculate the weight of KClO_3 ($W_3=W_2-W_1$).</p>	
<p>2. Start to heat the test tube in a diagonal position first gently, then more strongly. Heat the entire test tube to redness, and maintain the temperature for <u>fifteen minutes</u>. The mixture will first melt, then effervesce (produce gas) strongly, and finally solidify. DON'T KEEP OPEN SIDE OF THE TEST TUBE TOWARDS YOUR AND YOUR LAB-MATES FACES! Oxygen release can sputter very hot content as well! Move the test tube continuously on the flame, otherwise the glass may melt.</p>	
<p>3. Cool the test tube slowly and weigh (W_4).</p>	
<p>4. Heat the test tube and the contents to redness for additional <u>five minutes</u>. Cool and reweigh (W_4).</p>	
<p>5. Repeat Step 8 until your last weight will be the same with previous one. Your last weighing is W_f. Same weight means; you removed all of the oxygen from your compound. Calculate the weight of oxygen given off, W_{ox}. Calculate experimental percent oxygen by weight in KClO_3. Calculate the theoretical percent of oxygen in KClO_3. The atomic weights are as follows: O=16.0 g/mol; Cl=35.5 g/mol; K=39.1 g/mol. Calculate % Error as explained in theoretical part.</p>	

Part B: Analysis of a KClO_3 - KCl Sample

1. The composition of an unknown KClO_3 – KCl will be determined with the same procedure as in Part A. Take your unknown sample from your assistant. Follow the same procedure used in Part A with the unknown mixture instead of pure KClO_3 . Use the same notations (similar to Part A 1-11). Calculate the percent KClO_3 by weight in your unknown sample (see your data sheet).

Questions

- 1) How many kilograms of copper sulfide could be formed from the reaction of 2.70 mol of copper with excess sulfur?
- 2) Given that zinc chloride has a formula weight of 136.28 g/mol, what is its formula?
- 3) Calculate the percent composition of HNO_3 ? (H: 1 g/mol; N: 14 g/mol; O: 16 g/mol)

DATA SHEET

The Law of Definite Proportions

Student's Name :

Date:

Laboratory Section/Group No :

Assistant's Name and Signature:

A. Percent Oxygen in Potassium Chlorate

1. Weight of test tube and catalyst (W_1) g
2. Weight of test tube, catalyst and $KClO_3$ (W_2) g
3. Weight of $KClO_3$ ($W_2 - W_1 = W_3$) g
4. Weight of the test tube and the contents after first heating (W_4) g
5. Weight of the test tube and the contents after second heating (W_5) g
6. Weight of the test tube and the contents after third heating (W_6) g
7. Weight of the test tube and the contents after last heating (W_f) g
8. Weight of oxygen given off ($W_2 - W_f = W_{ox}$) g
9. Experimental % of oxygen [$(W_{ox} / W_3) \times 100$] %
10. Theoretical % of oxygen by weight in $KClO_3$ %
11. Percent error %

B. Analysis of a $KClO_3$ - KCl Sample

12. Weight of test tube and catalyst (W_1) g
13. Weight of test tube, catalyst and unknown (W_2) g
14. Weight of unknown ($W_2 - W_1 = W_3$) g
15. Weight of the test tube and the contents after first heating (W_4) g
16. Weight of the test tube and the contents after second heating (W_5) g
17. Weight of the test tube and the contents after third heating (W_6) g
18. Weight of the test tube and the contents after last heating (W_f) g
19. Weight of oxygen given off ($W_2 - W_f = W_{ox}$) g
20. Percent oxygen by weight in unknown %
21. % $KClO_3$ in sample %

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Experiment 3
Titration of Acids and Bases



Purpose: To become familiar with the techniques of titration, a volumetric method of analysis; to determine the molarity and pH of an acid solution.

Prelaboratory Work

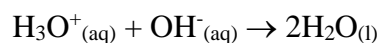
Before the experiment in the laboratory, you should be able to answer these questions.

1. What is the definition of standardization?
2. What is the definition of titration?
3. What is the definition of molarity?
4. Write the difference between equivalence points and end points.
5. Find the molarity of a solution that contains 3.78 g of $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ in 100 mL of solution?

Theory

According to Arrhenius, **acid** is a chemical substance which gives hydronium ion, H_3O^+ , when dissolved in water. Also, Bronsted-Lowry defined that acid acts as a proton donor. On the other hand, Arrhenius defined **base** in a way that it gives hydroxide ion, OH^- , when dissolved in water. According to Bronsted-Lowry, base acts as a proton acceptor.

One of the most common and familiar reactions in chemistry is the reaction of an acid with a base. This reaction is named as **neutralization** reaction, and the essential feature of this process in aqueous solution is the combination of hydronium ions with hydroxide ions to form water.



In this experiment you will perform this reaction to determine accurately the concentration of a sodium hydroxide solution that you have prepared. The process of determining the exact concentration (molarity) of a solution is called **standardization**. Next you will measure the concentration of the unknown acid solution. For this purpose, you are expected to measure the volume of your standard base that is required to exactly neutralize the unknown acid solution. The technique of accurately measuring the volume of a solution required to react with another reagent is termed **titration**.

During titration, you will use an **indicator** solution to understand whether you could neutralize your acid with a base or vice versa. Indicators change colors at different pH values. For example, phenolphthalein changes color from colorless to pink at a pH of about 9; in slightly more acidic solutions it is colorless, whereas, in more alkaline solutions it is pink. The color change is termed the **end point** of the titration.

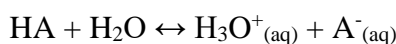
The point at which stoichiometrically equivalent quantities are brought together is known as the **equivalence point** of the titration. It should be noted that the equivalence point in a titration is a theoretical point.

Molarity (M) is used to define concentration of a solution more clearly, and it is defined as the number of moles of solute per liter of solution, or the number of millimoles of solute per milliliter of solution:

$$M = \frac{\text{moles.solute}}{\text{volume.of .solution}} = \frac{10^{-3} \text{ mole}}{10^{-3} \text{ liter}} = \frac{\text{mmol}}{\text{mL}} \quad [1]$$

Ph concept

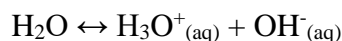
pH is a concept that is used to measure the acidity of a solution. It is related to concentration of H_3O^+ ion in molarity.



The concentration of $[\text{H}^+]$ may change over a wide range of values and these values are frequently expressed in terms of exponential numbers. For this reason, a simpler form of representation for $[\text{H}^+]$ is provided as follows: **pH = -log $[\text{H}^+]$**

There is another concept named as **pOH**. It is used to measure the basicity of the solution and related to concentration of OH^- in molarity: **pOH = -log $[\text{OH}^-]$**

When water is self ionized, hydronium and hydroxide ion formed in equal amounts:



$$K_w = [\text{H}^+][\text{OH}^-]$$

$$K_w = 1.0 \times 10^{-14} \text{ at } 25^\circ\text{C}$$

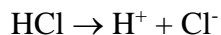
Therefore;

$$\text{pH} + \text{pOH} = 14$$

Example: What are the pH values for **a)** 0.1 M HCl and **b)** 0.1 M NaOH?

Solution:

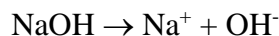
a) HCl is a strong acid, it dissociates almost completely in aqueous solution. Therefore 0.1 M HCl gives 0.1 M H^+ and 0.1 M Cl^- .



$$[H^+] = 0.1 \text{ M}$$

$$pH = -\log(0.1) = 1.00$$

b) NaOH is a strong base; it dissociates almost completely in aqueous solution. 0.1 M NaOH gives 0.1 M Na^+ and 0.1 M OH^- .



$$[OH^-] = 0.1 \text{ M}$$

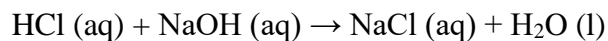
$$[H^+] = K_w / [OH^-] = 1 \times 10^{-14} / 0.1 = 1.0 \times 10^{-13} \text{ M.}$$

$$pH = -\log(1.0 \times 10^{-13}) = 13.00$$

or, $pH + pOH = 14$ $1 + pOH = 14$ then, $pOH = 13$

Strong Acid-Base Titration

As an example for neutralization reaction between strong acid (e.g. HCl) and a strong base (e.g. NaOH);



As a result, for a monoprotic acid and base at the end point;

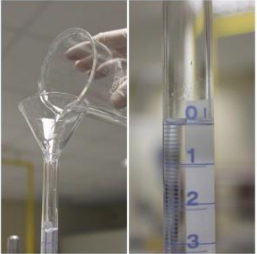


$$M_{\text{acid}} V_{\text{acid}} = M_{\text{base}} V_{\text{base}}$$

In this experiment, we use an acid-base indicator, phenolphthalein to determine the end point in the titration. We choose an indicator such that its color change occurs as closely as possible to the equivalence point.

Materials

Sodium hydroxide (NaOH)	600 mL beaker	Ring stand
Hydrochloric acid (HCl)	250 mL Erlenmeyer flasks	Buret clamp
Phenolphthalein solution	50 mL buret	Balance
Wash bottle	500 mL erlenmeyer flask	

Procedure

Part A: Standardization of Sodium Hydroxide Solution	
<p>1. Fill the buret with the NaOH solution and remove the air from the tip by running out some of the liquid into an empty beaker. Make sure that the lower part of the meniscus is at the zero mark or slightly lower.</p>	
<p>2. Take 15.00 mL of standard HCl solution into a clean Erlenmeyer flask and add a few drops of phenolphthalein solution.</p>	
<p>3. Start to add the sodium hydroxide solution slowly to your flasks of HCl solution while gently swirling the contents of the flask. As the sodium hydroxide solution is added, a pink color appears where the drops of the base come in contact with the solution. This coloration disappears with swirling. As the end point is approached, the color disappears more slowly, at which time the sodium hydroxide should be added drop by drop. The end point is reached when the colour of the solution turns from colorless to pink.</p>	
<p>4. Read the buret and record it. Repeat this procedure for two times. From the data you obtain in the three titrations, calculate the molarity of the sodium hydroxide solution to four significant figures.</p>	

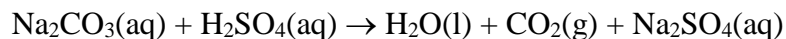
Part B: Analysis of an Unknown Acid

1. Obtain 15 mL of the unknown solutions. Add a few drops of phenolphthalein solution and titrate against the standard NaOH solution as in Part A.



Questions

1. Write the balanced chemical equation for the reaction of HCl with NaOH.
2. A solution of malonic acid, $\text{H}_2\text{C}_3\text{H}_2\text{O}_4$, was standardized by titration with 0.100 M NaOH solution. If 21.82 mL of the NaOH solution were required to neutralize completely 12.12 mL of the malonic acid solution, what is the molarity of the malonic acid solution?
3. Sodium carbonate is a reagent that may be used to standardize acids in the same way. In such standardization it was found that a 0.432-g sample of sodium carbonate required 22.3 mL of a sulfuric acid solution to reach the end point for the reaction.



What is the molarity of the H_2SO_4 ?

4. A solution contains 0.252 g of oxalic acid, $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$, in 500 mL. What is the molarity of this solution?

DATA SHEET
Titration of Acids and Bases

Student's Name : _____ Date: _____

Laboratory Section/Group No : _____

Assistant's Name and Signature : _____

A. Standardization of Sodium Hydroxide Solution

1. Molarity of HCl solution :
2. Volume of HCl solution:
3. Volume of NaOH solution used for the titrations 1st:2nd:3rd:
4. Molarity of NaOH solution:

B. Analysis of an Unknown Acid I

1. Volume of unknown acid solution:
2. Volume of NaOH solution used for the titration :
3. Molarity of unknown acid I:.....

C. Analysis of an Unknown Acid II

1. Volume of unknown acid solution:
2. Volume of NaOH solution used for the titration :
3. Molarity of unknown acid II:.....

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Experiment 4

Stoichiometry: The Reaction of Iron with Copper(II) Sulfate



Purpose: To enhance the understanding of stoichiometry, a reaction between iron and copper (II) sulfate (CuSO_4) solution will be conducted. This will help you to differentiate limiting and excess reactant in a chemical reaction. Finally the theoretical and percent yield of this reaction will be calculated.

Theory

Stoichiometry is the measurement of quantitative relationships in chemical formulas and equations.

Mostly in chemical reaction two or more reactants (reagents) are placed. The reagent that is totally consumed before other reactants at that time reaction stops defined as **limiting reagent**. Limiting reagent is the reactant, which is totally consumed before other reactants. The limiting reactant limits the

amount of product that can be formed since the consumption of the reactant stops the reaction. The reactant that is remained once the limiting reagent is consumed is called as **excess reagent**.

Example: How many moles of Ag are in 1.75 mol of Ag_2CrO_4 ?

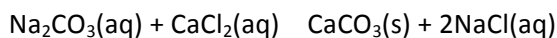
There are 2 moles of Ag atoms for each Ag_2CrO_4 formula unit

Therefore,

$$1.75 \text{ mol } \text{Ag}_2\text{CrO}_4 \times 2 = 3.5 \text{ mol Ag}$$

Example: Sodium carbonate, Na_2CO_3 , reacts with calcium chloride, CaCl_2 , to form calcium carbonate, CaCO_3 , and sodium chloride, NaCl . In an experiment 53 grams of Na_2CO_3 and 44.4 grams of CaCl_2 were mixed and 23.6 gr CaCO_3 was obtained. Which is the limiting reactant? What is the percent yield of CaCO_3 ?

(Molecular weight of $\text{Na}_2\text{CO}_3 = 106 \text{ g/mol}$, $\text{CaCl}_2 = 111 \text{ g/mol}$, $\text{CaCO}_3 = 100 \text{ g/mol}$ $\text{NaCl} = 58.5 \text{ g/mol}$)



Mole of Na_2CO_3 : $53 / 106 = 0.3$ moles of Na_2CO_3 are present.

Mole of CaCl_2 : $44.4 / 111 = 0.4$ moles of CaCl_2 are present.

1 mole of Na_2CO_3 reacts with 1 mole of CaCl_2 according to the above equation.

$$\frac{1 \text{ mole } \text{Na}_2\text{CO}_3}{0.3 \text{ mole } \text{Na}_2\text{CO}_3} = \frac{1 \text{ mole } \text{CaCl}_2}{x} \quad x = 0.3 \text{ moles of } \text{CaCl}_2 \text{ are needed.}$$

0.3 moles of CaCl_2 are needed and 0.4 moles of CaCl_2 are present. Therefore, CaCl_2 is excess reagent and Na_2CO_3 is limiting reactant.

Since Na_2CO_3 is limiting reactant it limits the amount of the product, CaCO_3 that will be formed.

$$\frac{1 \text{ mole Na}_2\text{CO}_3}{0.3 \text{ mole Na}_2\text{CO}_3} = \frac{1 \text{ mole CaCO}_3}{x} \quad x = 0.3 \text{ moles of CaCO}_3 \text{ will be formed}$$

$0.3 * 100 = 30$ gr of CaCO_3 will be formed. This amount is theoretical yield. Since the actual yield is the amount of product given, the percent yield will be calculated by the formula given below;

$$\text{Percent yield} = \frac{\text{actual yield}}{\text{theoretical yield}} \times 100$$

Then, percent yield for CaCO_3 is;

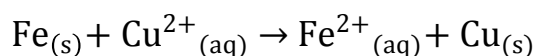
$$(23.6/30) * 100 = 78.7\%$$

In this experiment stoichiometric principles will be used to obtain the appropriate equation between the reaction of iron metal and copper (II) sulfate solution. When the reaction starts, the formation of metallic copper, which is precipitating during reaction, as a finely divided reddish-orange powder will be observed. This reaction is one of the example of single substitution reaction in which one element “displaces” with another element in a compound. The element which has ability of displacing other element from a compound is said to be “more active” than the displaced one. In this experiment, iron is more active than copper.

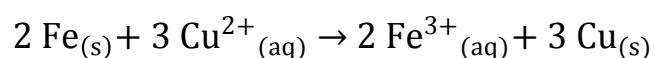
Two distinct forms of iron are present, namely Ferrous, Fe^{2+} and Ferric, Fe^{3+} . Stoichiometric principles will be used to determine which reaction is more dominant compared to other by examining the reaction between iron and copper (II) sulfate solution. If Fe^{2+} is formed, then **equation (1)** is dominant, but **equation (2)** will be selected if Fe^{3+} is formed. This can be determined by taking the mole ratio of copper

to iron. If the moles of copper are equal to the moles of iron, then equation (1) has taken place. If you obtain 1.5 moles of copper per mole of iron, in this case equation (2) should be selected Find out which equation is corresponding to the results of the experiment you have done.

Equation (1) $\text{Fe}_{(s)} + \text{CuSO}_{4(aq)} \rightarrow \text{FeSO}_{4(aq)} + \text{Cu}_{(s)}$, other representation is



Equation (2) $2 \text{Fe}_{(s)} + 3 \text{CuSO}_{4(aq)} \rightarrow \text{Fe}_2(\text{SO}_4)_3(aq) + 3 \text{Cu}_{(s)}$, other representation is

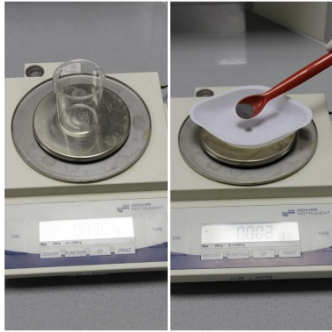
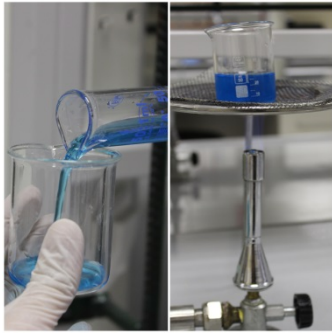
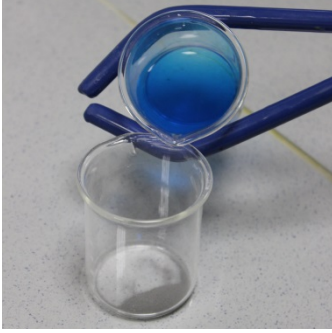


To the known amount of iron, excess of copper (II) sulfate solution will be added. The purpose of using excess solution is owing to provide the complete reaction of iron. The metallic copper produced will be weighed after washing and drying processes and these weightings will be used to calculate the moles of iron used and the moles of copper formed at the end of the reaction.

Materials

Fe powder	Acetone	Glass stick
Copper (II) Sulfate (CuSO_4)	Beaker	Bunsen burner

Procedure

The Reaction of Iron with Copper(II) Sulfate	
<p>1. Weigh a dry and clean 100 or 250 mL beaker and record the weight of it onto your data sheet. Then, accurately weigh 1.00 gram of iron powder into this beaker. Do not exceed 1.01 grams.</p>	
<p>2. Measure 30 mL of 1.0 M CuSO_4 solution by using a graduated cylinder. Pour this solution into another beaker, and heat gently to almost boiling.</p>	
<p>3. Slowly add hot CuSO_4 solution to the beaker that contains the iron powder. Stir the mixture a few times until completeness of the reaction. You should see copper forming. When the reaction has finished, allow the copper product to cool.</p>	

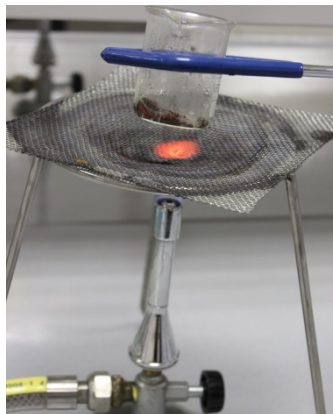
4. Then carefully decant the liquid from the copper into the waste container. Be careful not to lose any copper.



5. Add about 10 mL of distilled water to the solid copper and swirl to wash any remaining ions from the copper. Decant the wash water from the copper and add 10 more mL of distilled water, swirl and decant again. Wash copper particles finally with several mL of acetone (**CAUTION**-Acetone is very flammable). Swirl and allow standing a few minutes. Decant off the acetone.



6. The acetone readily dissolves the water and helps the removal of it from the medium. Swirl the beaker gently on low heat flame. Copper product should be spread in a single layer on the bottom of the beaker. Grinding of aggregates with a spatula makes the copper easy to dry. Be sure not to remove any copper from the beaker.



7. After drying, allow copper to cool and weigh the beaker plus copper to calculate the mass of copper formed. Record the mass on your data sheet. Finally, calculate the moles of iron used and the moles of copper formed to determine which reaction of iron is taking place, reaction (1) or reaction (2).



DATA SHEET

Stoichiometry: The Reaction of Iron with Copper(II) Sulfate

Student's Name : Date:

Laboratory Section/Group No :

Assistant's Name and Signature:

Data and Calculations

Mass of empty beaker :

Mass of iron used :

Moles of iron used :

Mass of beaker plus copper :

Mass of copper formed :

Moles of copper formed :

Moles of Cu divided by moles of Fe :

Reaction Equation :

Limiting Reagent :

Theoretical and Percent Yield :

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GENERAL CHEMISTRY

Experiment 5
**Molecular Weight Determination from
Freezing Point Depression**



Purpose: To learn colligative properties and to determine the molecular weight of sulfur by using one of the colligative properties.

Prelaboratory Work

Before the experiment in the laboratory, you should be able to answer these questions.

1. List the colligative properties.
2. What is the molality of a solution that contains 1.5 g of urea (molar mass = 60 g/mol) in 200 g of benzene?
3. Calculate the freezing point of a solution containing 5.85 g of NaCl in 200 g of water? (Na: 23 g/mol; Cl:35.5 g/mol)
4. A solution containing 1.00 g of an unknown substance in 12.5 g of naphthalene was found to freeze at 75.4°C. What is the molar mass of the unknown substance?
5. Define molality and molarity.

Theory

Some of the physical properties of solutions are independent of the nature of the solute and depend only upon the solute amount. These properties are called “colligative properties”. Vapor pressure lowering, Boiling point elevation, Freezing-point depression, and osmotic pressure are colligative properties.

Those effects introduced above are familiar to us in our daily life. In this experiment, freezing point depression property will be used. That’s why it is better to give examples or explanations related with this property.

Anti-freeze solution is used for lowering the freezing point of water in automobiles. (This solution is made by mixing water and ethylene glycol or water and methanol or water and another one.) When something is added to water, freezing point of water decreases automatically. The increasing addition amount decreases the freezing point more.

Another example is pouring salt onto the snow on the roads to prevent freezing in winter days. When salt is added to water, the resulting solution has lower freezing point (and also higher boiling point). Increasing the amount of salt decreases the freezing point more. Freezing point and boiling point of a solution differ from those of the pure solvent by amounts that are directly proportional to the molal concentration of the solute and can be expressed with the equation below:

$$\Delta T = K \cdot m$$

where ΔT is the freezing-point lowering or boiling-point elevation, K is molal freezing (or boiling) constant which is specific for each solvent, m is the molality of the solution (number of moles of solute /kg of solvent).

Some representative constants, boiling points, and freezing points are given in Table 1. For naphthalene, the solvent used in this experiment, the molal freezing constant (K_f) has a value of 6.90 °C/m.

TABLE 1. Molal Freezing Point and Boiling Point Constants

Solvent	Freezing point (°C)	K _{fp} (°C/m)	Boiling point (°C)	K _{bp} (°C/m)
CH ₃ COOH (acetic acid)	16.6	3.90	118.1	2.93
C ₆ H ₆ (benzene)	5.4	5.12	80.2	2.53
C ₂ H ₅ OH (ethyl alcohol)	-114.1	-	78.4	1.22
H ₂ O (water)	0.0	1.86	100.0	0.51
C ₁₀ H ₈ (naphthalene)	80.6	6.9	218	-

Example: What would be the freezing point of a solution containing 19.5 g of biphenyl (C₁₂H₁₀) dissolved in 100 g of naphthalene if the normal freezing point of naphthalene is 80.6°C?

Solution:

$$\text{moles of C}_{12}\text{H}_{10} = \frac{19.5 \text{ g}}{154 \text{ g/mol}} = 0.127 \text{ mol}$$

$$\frac{\text{moles of C}_{12}\text{H}_{10}}{\text{kg of naphthalene}} = \frac{0.127 \text{ mol}}{0.1 \text{ kg}} = 1.27 \text{ molal (m)}$$

$$\Delta T = (6.91 \text{ °C/m}) \times (1.27 \text{ m}) = 8.8 \text{ °C}$$

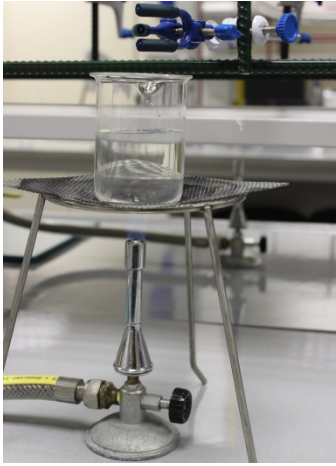
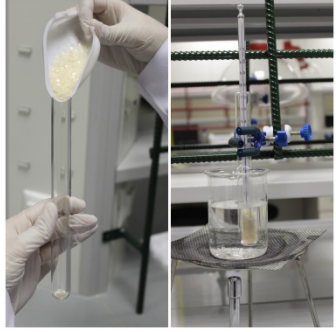
Since the freezing point is lowered, the observed freezing point of the solution will be

$$T_{\text{pure}} - T_{\text{solution}} = 80.6\text{°C} - 8.8\text{°C} = 71.8\text{°C}$$

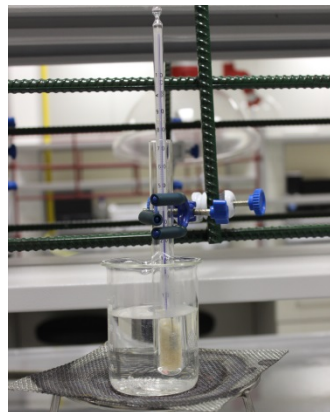
Materials

Naphthalene	Large test tube	600-mL beaker
Sulfur	Clamp	Bunsen burner
Thermometer	Ring and ring stand	

Procedure

PART A: Cooling Curve for Pure Naphthalene	
<p>1. Fill your 600-mL beaker nearly full of water and heat it to about 85 °C. Clamp the test tube in the water bath as shown in figure.</p>	
<p>2. Weigh 3 g of naphthalene and put the naphthalene in the test tube. Then insert the thermometer into the test tube-naphthalene as shown in figure above. Wait for naphthalene melts. When all of the naphthalene has melted, stop heating; remove the test tube out of water.</p>	

3. Starting with 85 °C; record temperature readings every 30 s. Shake the test tube slightly at every temperature reading. When the freezing point is reached, crystals will start to form, and the temperature will remain constant. After all the naphthalene freeze, temperature lowering will start again.

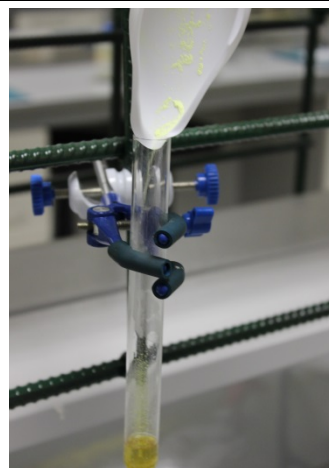


PART B: Determination of the Molar Mass of Sulfur

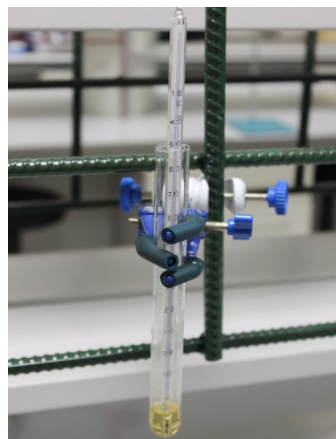
1. Weigh 0.5 g of sulfur.



2. Replace the test tube in the water bath and heat until all the naphthalene has melted. Add the sulfur into the test tube. Shake the test tube until all the sulfur has dissolved.



3. Remove the test tube out of the water bath, record the temperature every 30 s until all the naphthalene has solidified. Do not forget to shake the test tube at each temperature readings. Using the data above, draw two cooling graph and find ΔT .



DATA SHEET

Molecular Weight Determination from Freezing Point Depression

Student's Name :

Date:

Laboratory Section/Group No :

Assistant's Name and Signature:

1. Weight of Naphthalene:g

2. Weight of Sulfur:.....g

3. Freezing point of pure naphthalene, T_{pure} :..... °C

4. Freezing point of solution (naphth. + sulfur), T_{solution} :..... °C

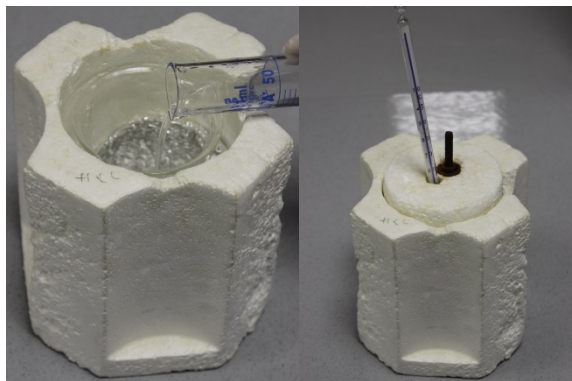
5. $\Delta T = T_{\text{pure}} - T_{\text{solution}} =$ °C

6. Molality of Sulfur:molal (m). (*Show calculations below..*)

7. Molar mass of unknown Sulfur:.....g/mol (*Show calculations below..*)

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GENERAL CHEMISTRY

Experiment 6
Heat of Neutralization



Purpose: To calculate enthalpy change of a reaction by using calorimeter and understand the difference between endothermic and exothermic reactions

Prelaboratory Work

Before the experiment in the laboratory, you should be able to answer these questions.

1. Calculate the volume of solution of 1.50 M HNO_3 required in part (A) of the procedure.
2. Calculate the mass of NH_4NO_3 required in part (B) of the procedure. Show your reasoning.
3. Explain in stepwise fashion (number the steps) how you will determine the ΔH of formation of NH_4NO_3 from your data and information given in the experiment. Use actual numbers whenever possible.
4. How many joules are required to change the temperature of 80.0 g of water from 23.3°C to 38.8°C ?

Theory

In chemical reactions, energy change is observed. This energy change is usually in the form of heat and at constant pressure it is defined as **heat of reaction** or **enthalpy change (ΔH)**. To form 1 mole of compound from its constituent elements, necessary amount of enthalpy change occurs and this change is defined as **enthalpy of formation**. If heat is released during the reaction, ΔH is shown with negative sign and the reaction is called **exothermic reaction**. If heat is absorbed during the reaction, ΔH is shown with positive sign and the reaction is called **endothermic reaction**.

Direct measurement of enthalpies of formation is difficult experimentally; so indirect methods involving enthalpies of reaction are used. Hess's Law states that the change in a thermodynamic property such as enthalpy depends on the initial and final states and is independent of path followed. An example for Hess's Law is given below.



Assume that ΔH_1 and ΔH_2 are known. If first and second reactions are added, net reaction becomes;



which is also formation reaction of $\text{NH}_3(\text{aq})$.

Given that $\Delta H_1 = -45.8 \text{ kJ/mol}$ and $\Delta H_2 = -35.4 \text{ kJ/mol}$, we can calculate the ΔH formation of $\text{NH}_3(\text{aq})$ as -81.2 kJ/mol .

The heat is measured experimentally by allowing the reaction to take place in a thermally insulated vessel called as **calorimeter**. If the calorimeter is perfectly insulated, no heat change occurs between system and surrounding and the system is defined as adiabatic ($Q=0$). Consequently, at constant pressure, ΔH_{system} is also equal to zero. The formulation of enthalpy change of the system, ΔH_{system} , is shown as in Equation (1).

$$\Delta H_{\text{system}} = \Delta T (\text{heat capacity of calorimeter} + \text{heat capacity of contents}) \quad (1)$$

For endothermic reaction in adiabatic system, Equation (1) can be written as below.

$$\Delta H_{\text{system}} = n\Delta H_{\text{reaction}} + C_p\Delta T$$

$$0 = n\Delta H_{\text{reaction}} + C_p\Delta T$$

$$n\Delta H_{\text{reaction}} = -C_p\Delta T$$

In a similar manner, for exothermic reaction in an adiabatic system, Equation (1) can be simplified as:

$$\Delta H_{\text{system}} = -n\Delta H_{\text{reaction}} + C_p\Delta T$$

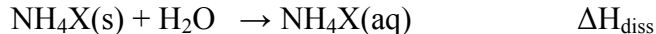
$$0 = -n\Delta H_{\text{reaction}} + C_p\Delta T$$

$$n\Delta H_{\text{reaction}} = C_p\Delta T$$

In this experiment, you will determine the heat of formation of various ammonium salts $\text{NH}_4\text{X}(\text{s})$ where X is Cl, NO_3 or SO_4 by combining measurements of the heat for the neutralization reaction;



And the heat of the dissolution reaction;


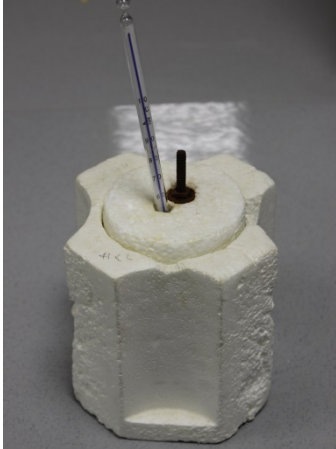
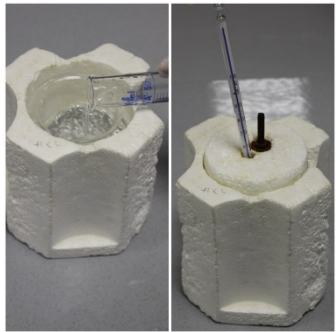


with known heats of formation of $\text{NH}_3(\text{aq})$ and $\text{HX}(\text{aq})$.

Materials

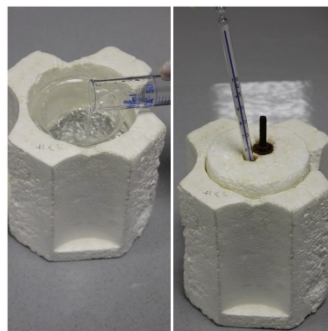
Nitric acid (HNO_3)	250 mL beaker	Graduated cylinder
Ammonia (NH_3)	Thermometers	
Ammonium nitrate (NH_4NO_3)	Styrofoam cups	

Procedure

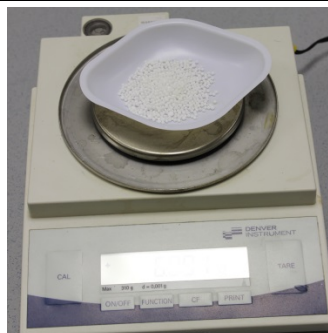
PART A: Heat of Neutralization	
<p>1. Obtain a Styrofoam cup. In the first cup, place 50 mL of 1.5 M NH_3.</p>	
<p>2. Place a thermometer in the cup containing the NH_3 and record temperature at 30 seconds intervals.</p>	
<p>3. Add the acid solution to the NH_3 and swirl to mix. Continue taking temperature data at 30 seconds intervals while swirling the solution occasionally.</p>	

PART B: Dissolving

1. Place a volume of distilled water equal to the final volume of solution from part (A) in a Styrofoam cup and record temperature data at 30 seconds intervals.



2. Weigh out that mass of NH_4NO_3 salt into a clean, dry beaker.



3. Immediately, add the weighed amount of salt, swirl to dissolve (use stirring rod if necessary), and continue taking temperature data at 30 seconds intervals.



Calculations

1. Plot temperature versus time graph using your data and determine ΔH_{neut} for (a) and ΔH_{diss} for (b).
2. Take ΔH_f of 1.5 M NH_3 as -81.2 kJ/mol and ΔH_f of 1.5 M HCl as -165.1 kJ/mol , calculate the ΔH_f of $\text{NH}_4\text{Cl}(s)$.
3. Do the same calculations for $\text{NH}_4\text{NO}_3(s)$ using -206.0 kJ/mol for the ΔH_f of 1.5 M HNO_3 .
4. Calculate ΔH_f of $(\text{NH}_4)_2\text{SO}_4$ using -884.2 kJ/mol for the ΔH_f of 1.5 M H_2SO_4 . [Note that all ΔH_f are per mol (not per 1.5 mol)]

DATA SHEET
Heat of Neutralization

Student's Name : _____ Date: _____

Laboratory Section/Group No : _____

Assistant's Name and Signature : _____

Prelaboratory Work

1. Volume of 1.5 M HNO₃ solution:

2. Mass of NH₄NO₃:

Time (s)	Temperature of NH ₃ solution in °C before adding 1.5 M HNO ₃	Temperature of distilled water in °C before NH ₄ NO ₃
0		
30		
60		
90		
120		
150		
180		
Time (s)	Temperature of NH ₃ + 1.5 M HNO ₃ solution in °C	Temperature of NH ₄ NO ₃ solution in °C
0		
30		
60		
90		
120		
150		
180		

Results

1. Calculate $\Delta H_{\text{neutralization}}$:

2. Calculate $\Delta H_{\text{dissociation}}$:

3. Calculate $\Delta H_{\text{formation}}$ of NH_4NO_3 :