

Faculty of Engineering
Chemical Engineering and Applied Chemistry



General Chemistry Laboratory Manual



ATILIM UNIVERSITY

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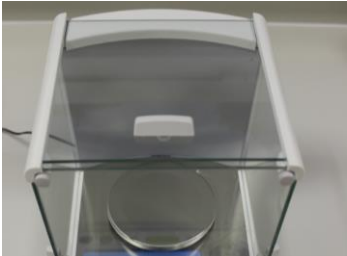
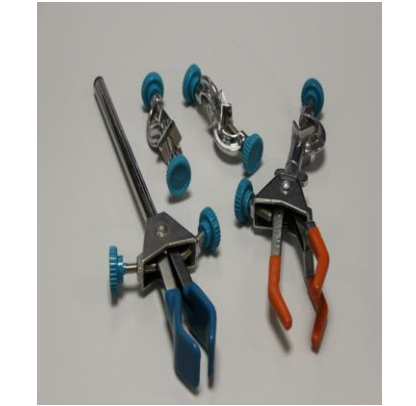


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GENERAL CHEMISTRY LABORATORY RULES

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

		
Beakers	Erlenmeyer Flask	Graduated Cylinder
		
Pisette	Thermometer	Burette
		

<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	

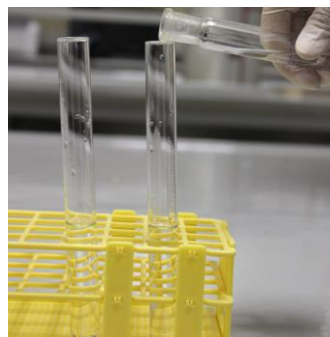
Procedure

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.

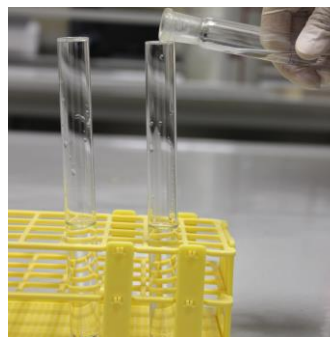


2. Take small amount (approximately half of a pea) of sodium carbonate; Na_2CO_3 , and starch and place them into test tubes that contain water. Shake the tubes gently and observe whether or not the substances dissolve. Some substances dissolve slowly, in this case set the test tube aside for few minutes 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 graduated 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 (KMnO₄) 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 ₂ CO 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

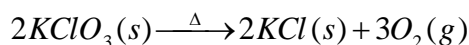
The law of definite proportions states that a chemical compound always contains exactly the same **proportion** of elements by mass. Law of definite proportions shows a good way to find percent weight or exact weight of a desired element in a compound. It also gives useful information to find empirical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

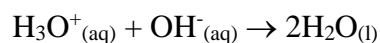
Before the experiment in the laboratory, you should be able to answer these questions.

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.

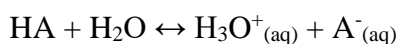
The point at which stoichiometrically equivalent quantities are brought together is known as the **equivalence point** of the titration. It should be noted that the equivalence point in a titration is a theoretical point.

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

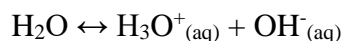
pH is a concept that is used to measure the acidity of a solution. It is related to concentration of H_3O^+ ion in molarity.



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:



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

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

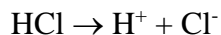
Therefore;

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

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

Solution:

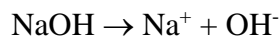
a) HCl is a strong acid, it dissociates almost completely in aqueous solution. Therefore 0.1 M HCl gives 0.1 M H^+ and 0.1 M Cl^- .



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

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

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^- .



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

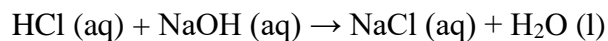
$$[H^+] = K_w / [OH^-] = 1 \times 10^{-14} / 0.1 = 1.0 \times 10^{-13} \text{ M.}$$

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

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;

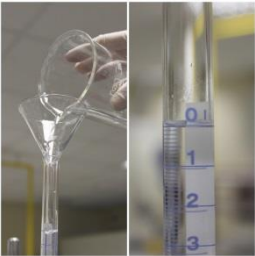


$$M_{\text{acid}} V_{\text{acid}} = M_{\text{base}} V_{\text{base}}$$

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>	

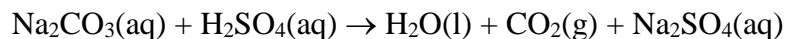
Part B: Analysis of an Unknown Acid

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:.....