

## CONTENTS

Introduction	
Safety Rules	
Laboratory Equipment	
Periodic Table	
Basic Laboratory Techniques	1
Conservation of Matter	9
Stoichiometry	13
Formula and Composition of a Hydrate	17
Determination of Heat of Fusion	22
Preparation of $\text{K}_3[\text{Fe}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$ and % Yield	28
Equivalent Weight of a Metal	33
Oxidation-Reduction Reactions	39
Soap	44
Blue Bottle Reaction	49
Solutions	53
Effect of Concentration on Reaction Rate	59
Effect of Temperature on Reaction Rate	66
Effect of Type of Matter on Reaction Rate	71
Acid-Base Titrations	75
pH and Indicators	80
Titration of Vinegar	85
Chemical Equilibrium	90
Determination of Water Hardness	96
References	102

## INTRODUCTION

The chemistry laboratory is used to teach the student about chemistry and scientific techniques. Your work in the laboratory will be one of the most important phases of your study of chemistry. All of the facts, principles, theories and laws that you will meet with in chemistry area based on observations and experiments. These experiments are the foundations of the science of chemistry. As a science student you should do your work sincerely and objectively.

The experiments you will perform are designed to do four things: (1) to show you how the facts and laws of chemistry have been determined and in so doing introduce you to the so-called “scientific method” of doing things, (2) to teach you certain facts, principles and laws, (3) to teach you to apply certain facts and principles in answering specific questions and solving problems, (4) to teach you specific laboratory techniques.

The amount of chemicals used has been minimized to increase safety and reduce the amount of waste and pollution. No carcinogenic and toxic metal salts are used in the experiments. The experiments have been selected to minimize hazards and costs. It should be kept in mind that it is impossible to remove every potential danger from a chemistry laboratory. You should follow directions, obey the safety rules and no experiment should be conducted alone in the laboratory.

Chemistry plays an extremely important role in today’s society. This laboratory work will be your first step into the world of chemistry. Begin your experience with a positive attitude and you will find that chemistry is an exciting and enjoyable adventure.

Good luck and best wishes for an error- free and accident-free term.

## **LABORATORY SAFETY RULES**

The chemistry laboratory can be a place of discovery and learning. However, by the very nature of laboratory work, it can be a place of danger if proper common-sense precautions aren't taken. While every effort has been made to eliminate the use of explosive, highly toxic, and carcinogenic substances from the experiments which you will perform, there is a certain unavoidable hazard associated with the use of a variety of chemicals and glassware. You are expected to learn and adhere to the following general safety guidelines to ensure a safe laboratory environment for both yourself and the people you may be working near. Additional safety precautions will be announced in class prior to experiments where a potential danger exists. Students who fail to follow all safety rules may be asked to leave the lab or suffer grading penalties.

### **SAFETY IS FIRST!**

- 1.** Do not perform unauthorized experiments or work in a laboratory alone.
- 2.** Approved eye protection must be worn at all times in the laboratory. Eye protection must be splash proof chemical goggles and be approved by your instructor. If you do get a chemical in your eye rinse immediately with large quantities of water using the eye-wash stations.
- 3.** Long hair and loose clothing must be confined while in a laboratory.
- 4.** All students must wear lab coats at all times. Your legs must be completely covered below the knee by your lab coat.
- 5.** Closed shoes with socks must be worn at all times – open-toed shoes, backless shoes, sling backs, clogs, and sandals are not permitted.
- 6.** Know the location and proper use of fire extinguishers, fire blankets, safety showers, eye wash devices, and first aid kits.
- 7.** Before obtaining any chemicals carefully read the label on the reagent bottles.
- 8.** Eating, smoking, chewing and drinking are not allowed in a chemistry laboratory.
- 9.** Thoroughly wash your hands after leaving the laboratory.
- 10.** Use the fume hoods when toxic or irritating vapors are involved.

- 11.** Mouth suction is never used to fill a pipette.
- 12.** Never force glass tubing through cork or rubber stoppers without proper lubrication.
- 13.** Never direct the open end of test tube toward yourself or anyone else.
- 14.** Never pour water into concentrated acid.
- 15.** Learn the proper procedure for igniting and operating a laboratory burner. Always extinguish the flame when the burner is not being used. Make sure that all flammable reagents are well removed before lighting the burner.
- 16.** Liquid and solid waste containers must be properly used at all times.
- 17.** Never place chemicals directly on the balance pan. Always use a proper weighing container when using a balance to weigh a chemical. Never pour chemicals directly over the balance.
- 18.** Never return unused chemicals to their original container (unless directed to do so by the instructor).
- 19.** Securely replace lids, caps, and stoppers after removing reagents from containers.
- 20.** Always wipe spatulas clean before and after inserting into reagent bottles.
- 21.** Report any accident and/or injury, however minor, to your instructor immediately.
- 22.** Never place anything that is not directly required for the experiment on laboratory desks; other items may interfere with the experiment.
- 23.** All personal belongings should be placed in the bookcases as you enter the laboratory.
- 24.** Clean up any spill immediately.
- 25.** Before leaving the laboratory, make sure your work area is clean and dry. Ensure that all gas, water, vacuum, and air valves are completely turned off.
- 26.** Your instructor is available for any assistance you may need. Never hesitate to ask questions especially if there is any question concerning proper operating procedure. Be sure that you understand every instruction before proceeding.

**27.** In any emergency, to get the lab supervisor attention is to SCREAM!

**28.** Absolutely no horseplay will be tolerated in laboratory.

**29.** If an acid is to be diluted, pour acid slowly into the water with constant stirring. Never add water to acid.

**30.** Students should use their own calculator. Mobile phones cannot be used as calculator.

**31.** Be familiar with the following terms and their effects.

Flammable:      They burn.

Irritants      :      They irritate eyes, lungs and skin.

Toxic           :      They are poisonous, effective either the short or long term.

Carcinogenic:      They cause cancer.

Teratogenic   :      They cause defects in the unborn fetus.

Mutagenic      :      They cause genetic mutations.

Explosive       :      They explode, usually on being mixed with air.

Corrosive       :      They burn the eyes, lungs and skin.

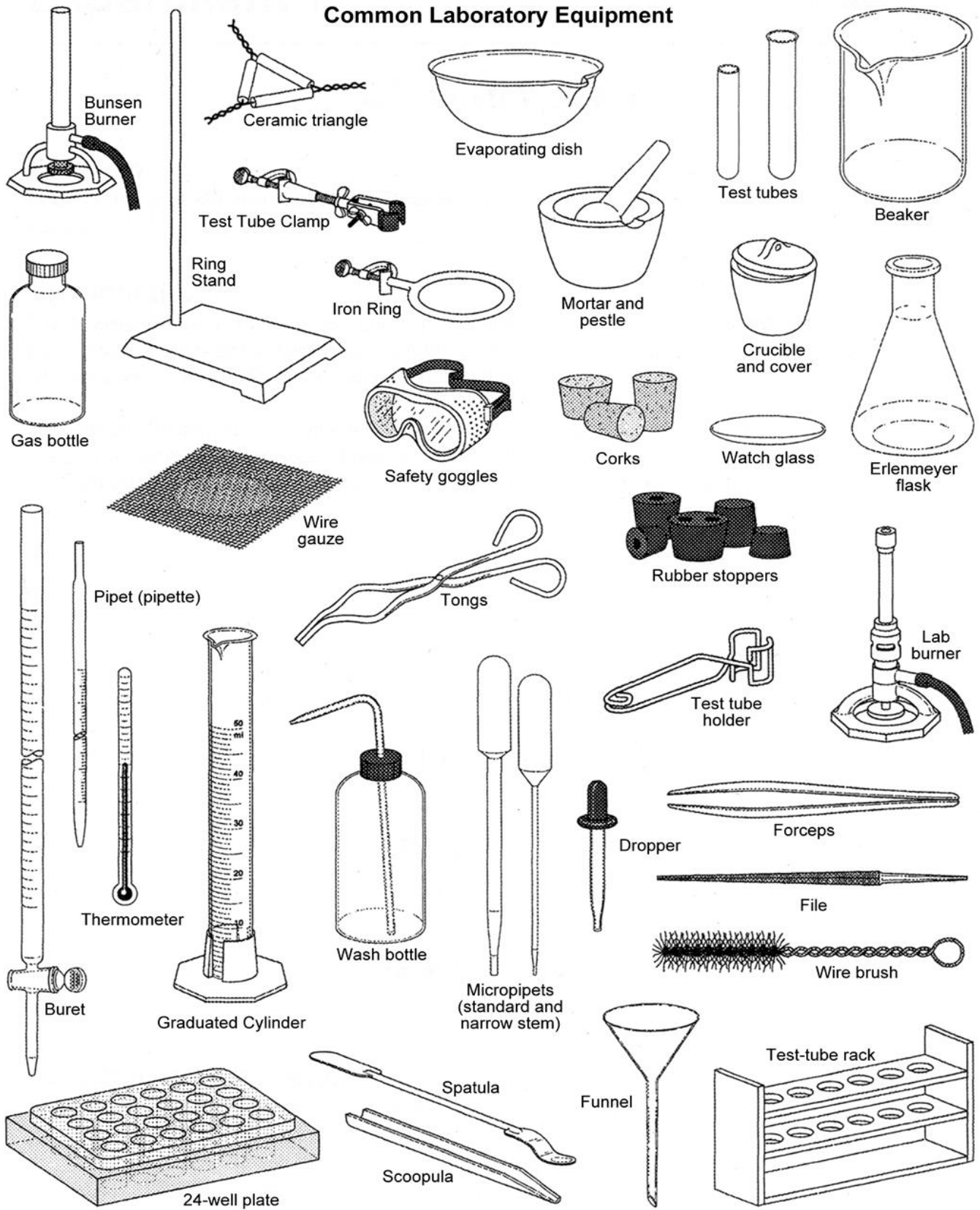
*This is to certify that I have read and that I understand this paper entitled “Laboratory Safety Rules”. I certify that I will obey each and every rule stated in the paper and will adhere to each of them in my chemistry courses. I also understand that if I fail to abide by the safety rules, I am doing so at my own risk and will not hold Marmara University or laboratory instructor liable for any injuries that result.*

*Name:*

*Date:*

*Signature:*

## Common Laboratory Equipment



# PERIODIC TABLE OF THE ELEMENTS

<http://www.ktf-split.hr/periodni/en/>

GROUP	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
PERIOD	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
1	<b>H</b> 1.0079 HYDROGEN																	<b>He</b> 4.0026 HELIUM
2	<b>Li</b> 6.941 LITHIUM	<b>Be</b> 9.0122 BERYLLIUM																
3	<b>Na</b> 22.990 SODIUM	<b>Mg</b> 24.305 MAGNESIUM																
4	<b>K</b> 39.098 POTASSIUM	<b>Ca</b> 40.078 CALCIUM	<b>Sc</b> 44.956 SCANDIUM	<b>Ti</b> 47.867 TITANIUM	<b>V</b> 50.942 VANADIUM	<b>Cr</b> 51.996 CHROMIUM	<b>Mn</b> 54.938 MANGANESE	<b>Fe</b> 55.845 IRON	<b>Co</b> 58.933 COBALT	<b>Ni</b> 58.693 NICKEL	<b>Cu</b> 63.546 COPPER	<b>Zn</b> 65.39 ZINC	<b>Al</b> 26.982 ALUMINIUM	<b>Si</b> 28.086 SILICON	<b>P</b> 30.974 PHOSPHORUS	<b>S</b> 32.065 SULPHUR	<b>Cl</b> 35.453 CHLORINE	<b>Ar</b> 39.948 ARGON
5	<b>Rb</b> 85.468 RUBIDIUM	<b>Sr</b> 87.62 STRONTIUM	<b>Y</b> 88.906 YTRIUM	<b>Zr</b> 91.224 ZIRCONIUM	<b>Nb</b> 92.906 NIOBIUM	<b>Mo</b> 95.94 MOLYBDENUM	<b>Tc</b> (98) TECHNETIUM	<b>Ru</b> 101.07 RUTHENIUM	<b>Rh</b> 102.91 RHODIUM	<b>Pd</b> 106.42 PALLADIUM	<b>Ag</b> 107.87 SILVER	<b>Cd</b> 112.41 CADMIUM	<b>In</b> 114.82 INDIUM	<b>Sn</b> 118.71 TIN	<b>Sb</b> 121.76 ANTIMONY	<b>Te</b> 127.60 TELLURIUM	<b>I</b> 126.90 IODINE	<b>Xe</b> 131.29 XENON
6	<b>Cs</b> 132.91 CAESIUM	<b>Ba</b> 137.33 BARIUM	<b>La-Lu</b> 57-71 Lanthanide	<b>Hf</b> 178.49 HAFNIUM	<b>Ta</b> 180.95 TANTALUM	<b>W</b> 183.84 TUNGSTEN	<b>Re</b> 186.21 RHENIUM	<b>Os</b> 190.23 OSMIUM	<b>Ir</b> 192.22 IRIDIUM	<b>Pt</b> 195.08 PLATINUM	<b>Au</b> 196.97 GOLD	<b>Hg</b> 200.59 MERCURY	<b>Tl</b> 204.38 THALLIUM	<b>Pb</b> 207.2 LEAD	<b>Bi</b> 208.98 BISMUTH	<b>Po</b> (209) POLONIUM	<b>At</b> (210) ASTATINE	<b>Rn</b> (222) RADON
7	<b>Fr</b> (223) FRANCIUM	<b>Ra</b> (226) RADIUM	<b>Ac-Lr</b> 89-103 Actinide	<b>Rf</b> 104 (261) RUTHERFORDIUM	<b>Db</b> 105 (262) DUBNIUM	<b>Sg</b> 106 (266) SEABORGIUM	<b>Bh</b> 107 (264) BOHRNIUM	<b>Hs</b> 108 (277) HASSIUM	<b>Mt</b> 109 (268) MEITNERIUM	<b>Uun</b> 110 (281) UNUNNIUM	<b>Uuu</b> 111 (272) UNUNUNIUM	<b>Uub</b> 112 (285) UNUNBIUM						

(1) Pure Appl. Chem., 73, No. 4, 667-683 (2001)

Relative atomic mass is shown with five significant figures. For elements with no stable nuclides, the value enclosed in brackets indicates the mass number of the longest-lived isotope of the element.

However three such elements (Th, Pa, and U) do have a characteristic terrestrial isotopic composition, and for these an atomic weight is tabulated.

Editor: Aditya Vardhan (adivar@netlinx.com)

## LANTHANIDE

57 138.91 <b>La</b> LANTHANUM	58 140.12 <b>Ce</b> CERIUM	59 140.91 <b>Pr</b> PRASEODYMIUM	60 144.24 <b>Nd</b> NEODYMIUM	61 (145) <b>Pm</b> PROMETHIUM	62 150.36 <b>Sm</b> SAMARIUM	63 151.96 <b>Eu</b> EUROPIUM	64 157.25 <b>Gd</b> GADOLINIUM	65 158.93 <b>Tb</b> TERBIUM	66 162.50 <b>Dy</b> DYSPROSIUM	67 164.93 <b>Ho</b> HOLMIUM	68 167.26 <b>Er</b> ERBIUM	69 168.93 <b>Tm</b> THULIUM	70 173.04 <b>Yb</b> YTTERIUM	71 174.97 <b>Lu</b> LUTETIUM
-------------------------------------	----------------------------------	--	-------------------------------------	-------------------------------------	------------------------------------	------------------------------------	--------------------------------------	-----------------------------------	--------------------------------------	-----------------------------------	----------------------------------	-----------------------------------	------------------------------------	------------------------------------

## ACTINIDE

89 (227) <b>Ac</b> ACTINIUM	90 232.04 <b>Th</b> THORIUM	91 231.04 <b>Pa</b> PROTACTINIUM	92 238.03 <b>U</b> URANIUM	93 (237) <b>Np</b> NEPTUNIUM	94 (244) <b>Pu</b> PLUTONIUM	95 (243) <b>Am</b> AMERICIUM	96 (247) <b>Cm</b> CURIUM	97 (247) <b>Bk</b> BERKELIUM	98 (251) <b>Cf</b> CALIFORNIUM	99 (252) <b>Es</b> EINSTEINIUM	100 (257) <b>Fm</b> FERMIUM	101 (258) <b>Md</b> MEDELEVIIUM	102 (259) <b>No</b> NOBELIUM	103 (262) <b>Lr</b> LAWRENCIUM
-----------------------------------	-----------------------------------	--	----------------------------------	------------------------------------	------------------------------------	------------------------------------	---------------------------------	------------------------------------	--------------------------------------	--------------------------------------	-----------------------------------	---------------------------------------	------------------------------------	--------------------------------------

Copyright © 1998-2003 EniG (eni@ktf-split.hr)

## **EXPERIMENT NO: 1      BASIC LABORATORY TECHNIQUES**

### **Chemicals and Apparatus**

- \*Potassium Permanganate,  $\text{KMnO}_4$
- \*Barium Chloride,  $\text{CaCl}_2$
- \*Sulfuric Acid,  $\text{H}_2\text{SO}_4$
- \*Potassium Chloride,  $\text{KCl}$
- \*Potassium Nitrate,  $\text{KNO}_3$
- \*Copper Nitrate,  $\text{Cu}(\text{NO}_3)_2$
- \*Test Tube, Pipet
- \*Distillation Apparatus
- \*Bunsen Burner or Hotplate
- \*Clamp and rings

### **PURPOSE**

The purpose of this experiment is to learn the use of some basic laboratory techniques that depends on physical properties of substances. In the first part of this experiment the students will receive instructions for the use of a balance and will perform a simple weighing exercise. In the second part, students will construct a simple laboratory apparatus and gain experience about some methods by which chemical substances may be separated such as distillation, crystallization and decantation.

### **THEORY**

A physical property is a characteristic that can be observed for a material without changing its chemical identity. A chemical property is a characteristic of a material involving its chemical change. A mixture is a material that can be separated by physical means. Distillation is a physical process in which an easily evaporized (volatile) liquid is separated from an another substance by evaporating and condensing. Thus, it is used to separate substances of different volatilities, different boiling points. In part A, water will be separated



from potassium permanganate solution 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.

Solubility is another physical property of substances. It is the amount of a substance that dissolves in a given quantity of solvent at a given temperature. It depends on both the substance and the solvent. The solubility of substances in one another varies widely. A factor that can limit solubility is the relative forces of attraction between the species. There is a general rule “like dissolves like”. Usually substances have limited solubility in one another. If the amount of substance present in the given solvent is greater than the solubility value precipitation occurs. Decantation is simply pouring off the liquid over a precipitate. In part B, how to form a precipitate and what decantation means will be studied.

In part C, separation of two water soluble salts on the basis of their different solubilities at room temperature will be studied. In this part remember that  $\text{Cu}(\text{NO}_3)_2$  is blue colored and  $\text{KNO}_3$  is white.

## I. Measuring Mass

The laboratory balance is the most commonly used and abused equipment in General Chemistry Laboratory. To keep it in good working order, you have to follow the rules given below:

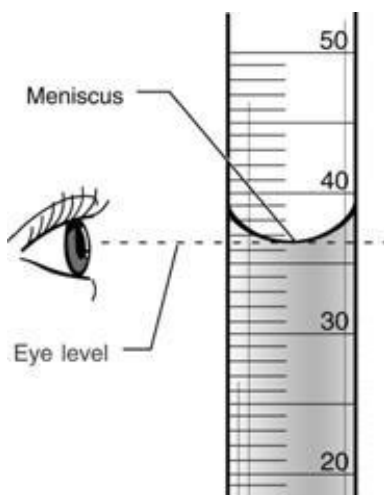


- Handle balances with care, they are expensive.
- Do not place chemicals directly on the pan; use a container (weighing paper, watch glass, beaker, test tube etc.)
- Do not drop anything on the pan.
- If it is not operating correctly, see your instructor. **DO NOT TRY TO FIX IT YOURSELF**
- Hot objects should never be placed on a balance.
- After weighing your sample, set everything back to zero.
- Clean the balance of any spilled chemicals.

## II. Measuring Volumes

Different glassware can be used to measure the volume of a liquid. Some of them give a rough result, like a graduated cylinder, but some measure an exact volume. Burets and pipets are used to measure exact volumes of liquids.

The volume of a liquid is read **at the bottom of its meniscus**, no matter which one is used. You should keep your eye at the same level with the meniscus (not above or below), so the volume can be read exactly. A clear liquid is read more easily than a colored one. If the solution has a dark color, since the meniscus can not be seen, you should read it from its upper level.



If a pipet is used to measure the volume, a rubber bulb should be used to suck the solution in. **DO NOT USE YOUR MOUTH; DO NOT SUCK IN ANY SOLUTION, EVEN IF IT IS WATER!**

## PROCEDURE

### A. Distillation

First a simple distillation apparatus must be constructed as shown in the figure.

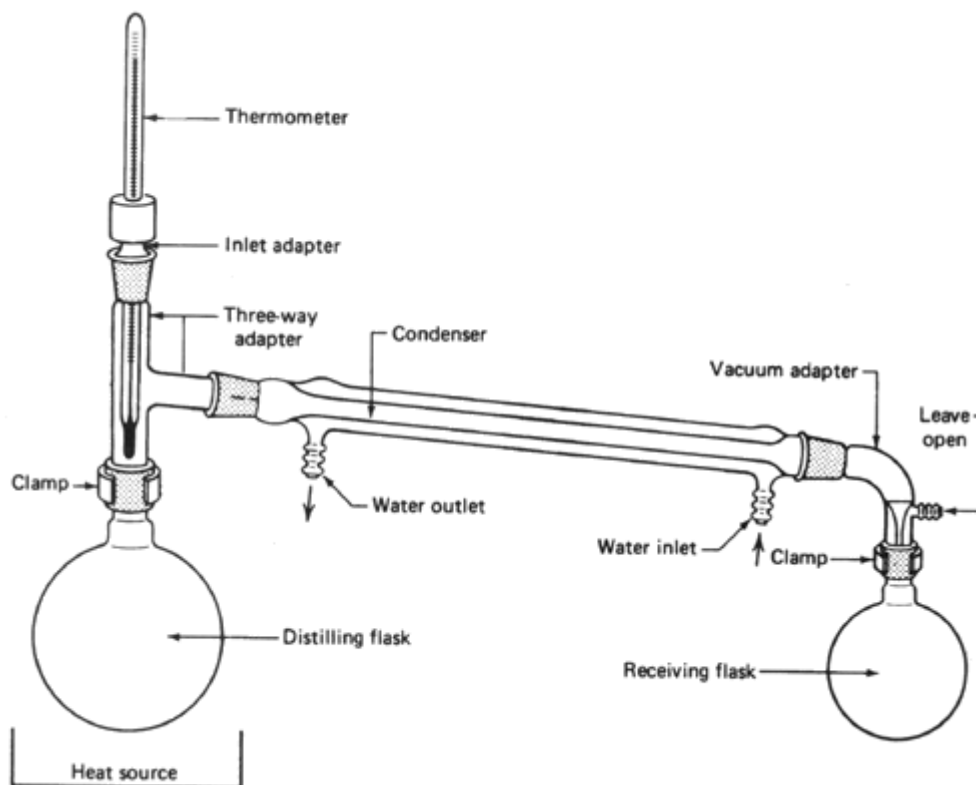


Figure 1. Distillation Apparatus

Pour 20 mL potassium permanganate solution (solid  $\text{KMnO}_4$  dissolved in water) into 100 mL distilling flask. Light the Bunsen burner or turn on the hotplate (your instructor will show how to use Bunsen burner or hotplate). Heat the  $\text{KMnO}_4$  solution until about 10 mL of liquid have been distilled into the receiving flask. Observe the differences in color between the distillate and the color of the  $\text{KMnO}_4$  solution before and after distillation. Write your observations on the report sheet. Waste  $\text{KMnO}_4$  solution should be discarded in the waste box. Pour the left over to the storage bottle.

## B. Precipitation of Barium Sulfate, $\text{BaSO}_4$

Mix 5 mL of barium chloride ( $\text{BaCl}_2$ ) solution with 5 mL of dilute sulphuric acid ( $\text{H}_2\text{SO}_4$ ) solution and observe what happens. Write your observation on report sheet (ex: what color of the precipitate?). Allow the contents of the test tube to stand until all solid  $\text{BaSO}_4$  has settled down. After the settlement is complete, you will be able to see the clear solution on the top of the precipitate; pour off only the liquid part into a beaker. Separation of a solid and liquid by pouring off the liquid is called **decantation**. Clean your tube.

## C. Recrystallization

Put about 5 g of potassium nitrate ( $\text{KNO}_3$ ) - copper nitrate ( $\text{Cu}(\text{NO}_3)_2$ ) mixture in a beaker and add 10 ml of distilled water. Heat the beaker gently from the sides by changing the position of the beaker frequently until all the solid has dissolved. Cool the beaker to room temperature and then cool the solution in beaker by passing cold tap water from outside of the beaker until no more crystals appear to form. Filter the solution (**Your instructor will show you how to use filter paper and funnel**). Save the filtrate (solution that passes through a filter). Wash the solid once or twice with a small amount of cold water. Observe the color of the solid and the filtrate (what are the identity and the colors of the solid and the solution?). Pour the crystals and the solution into the container labeled as " $\text{Cu}(\text{NO}_3)_2$ -  $\text{KNO}_3$  mixture".

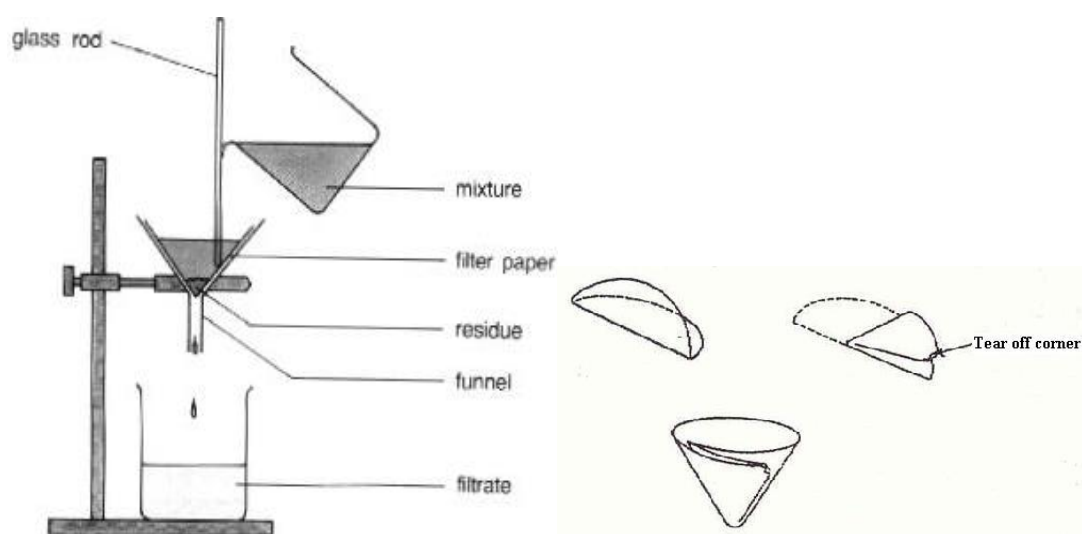


Figure 2. Filtration Process

# HAZARDOUS MATERIALS CLASSIFICATION



Chemicals are labeled according to National Fire Protection Association standards which describe the four possible hazards of a chemical and give a numerical rating from “0” to “4”.

## Report Sheet A

Student's Name:

Lab. Instructor name & sign. :

Lab. Section :

Date :

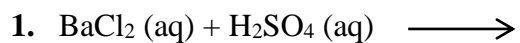
---

Name of the Experiment:

### A. Separation by Distillation

- a. Color of the solution before distillation :
- b. Color of the solution after distillation :
- c. Color of the distillate :

### B. Precipitation of Barium Sulfate, BaSO<sub>4</sub>



2. Color of the precipitate :

### C. Recrystallization

- a. Color of the solid :
- b. Color of the filtrate :

**Report Sheet B** (*to be submitted to the instructor*)

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

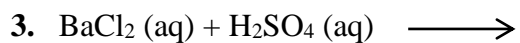
**D. Separation by Distillation**

**d.** Color of the solution before distillation :

**e.** Color of the solution after distillation :

**f.** Color of the distillate :

**E. Precipitation of Barium Sulfate, BaSO<sub>4</sub>**



**4.** Color of the precipitate :

**F. Recrystallization**

**c.** Color of the solid :

**d.** Color of the filtrate :

## **EXPERIMENT NO: 2      CONSERVATION OF MATTER**

### **Chemicals and Apparatus**

\*Sodium carbonate,  $\text{Na}_2\text{CO}_3$

\*Calcium Chloride,  $\text{CaCl}_2$

\*Sulfuric Acid,  $\text{H}_2\text{SO}_4$

\*Beakers

### **PURPOSE**

In this experiment the student will test the law of Conservation of Matter. This will be done by comparing the moles of a particular product with moles of one of the reactants using stoichiometry calculations.

### **THEORY**

The law of conservation of mass/matter, also known as law of mass/matter conservation (or the Lavoisier law), states that the mass of a closed system will remain constant, regardless of the processes acting inside the system. An equivalent statement is that matter cannot be created/destroyed, although it may be rearranged. This implies that for any chemical process in a closed system, the mass of the reactants must equal the mass of the products. In the study of mass changes in chemical reactions, it is of fundamental importance to find out whether the total mass present is the same before and after reaction.



## PROCEDURE

Label three of 50 mL beakers. In to each of three beakers pour the solutions as indicated below:

Beaker 1: 10 mL of the solution of sodium carbonate

Beaker 2: 3 mL of the solution of calcium chloride

Beaker 3: 3 mL of the solution of sulfuric acid

Make sure that the exteriors of the beakers are dry. Place all three beakers simultaneously on an analytical balance, weigh and record the total mass.

Removing the beakers from the balance case to protect the balance, carefully pour the solution of calcium chloride into the beaker containing the solution of sodium carbonate. Swirl gently to mix. Note any changes that occur. Set three of them back on the balance case and redetermine the total mass.

Remove the beakers again from the balance case and add sulfuric acid into the mixture of solutions 1 and 2 carefully. Swirl and note any evidence of reaction. Continue to swirl until all evidence of reaction has disappeared. If the beaker is not at room temperature (you can determine by touching it with inside of your wrist) wait a few minutes until it reaches to room temperature. Finally, place three of the beakers on the balance and redetermine the total mass.

*Who is Antoine Lavoisier?*

*Antoine-Laurent de Lavoisier (also Antoine Lavoisier after the French Revolution; 26 August 1743 – 8 May 1794) the "father of modern chemistry" was a French nobleman prominent in the histories of chemistry and biology. He named both oxygen (1778) and hydrogen (1783) and helped construct the metric system, put together the first extensive list of elements, and helped to reform chemical nomenclature. He was also the first to establish that sulfur was an element (1777) rather than a compound. He discovered that, although matter may change its form or shape, its mass always remains the same. He was an administrator of the Ferme Générale and a powerful member of a number of other aristocratic councils. All of these political and economic activities enabled him to fund his scientific research. At the height of the French Revolution, he was accused by Jean-Paul Marat of selling watered-down tobacco, and of other crimes and was eventually guillotined a year after Marat's death.*

## Report Sheet A

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

Total mass before first mixing =

Total mass after first mixing =

Total mass before second mixing =

Write down the chemical equations for the reactions occurred:

**Question:**

1. What do your data indicate about the mass change in the chemical reactions?

**Report Sheet B** (*to be submitted to the instructor*)

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

Total mass before first mixing =

Total mass after first mixing =

Total mass before second mixing =

Write down the chemical equations for the reactions occurred:

**Question:**

1. What do your data indicate about the mass change in the chemical reactions?

## EXPERIMENT NO: 3      STOICHIOMETRY

### Chemicals and Apparatus

\*Potassium chlorate,  $\text{KClO}_3$

\*Manganese (IV) oxide,  $\text{MnO}_2$  or Iron (III) Oxide,  $\text{Fe}_2\text{O}_3$

\*Test Tube or Curricible

### PURPOSE

This experiment is about calculating the percent oxygen in Potassium Chlorate. The experiment also gives student an experience in gravimetric analysis (analysis by weighing).

### THEORY

Stoichiometry is the mathematics behind the science of chemistry. Using known physical laws such as the law of conservation of mass and the law of definite proportions, stoichiometry is used to gather information about the amounts of various elements used in a chemical reaction, and whether they took the form of gases, solids, or liquids. Using mathematics, someone can determine the quantities of particular elements needed to create a desired reaction, or the quantities used in the generation of a reaction which has already occurred.

The law of definite proportions states that a pure, always contains the same combined in the same proportions by mass, regardless of the origin or mode of preparation of the compound. Since the atoms of each element can be assigned a definite average weight, the composition by weight of a given compound is some definite value fixed by the atomic weight and the molecular formula.

The mass percent of an element B in a compound  $\text{M}_a\text{X}_b$  can be calculated by the following equation:

$$\text{Mass \% of X} = \frac{b. (\text{Atomic weight of X})}{\text{Formula weight of } \text{M}_a\text{X}_b} \cdot 100$$

## PROCEDURE

In a clean, dry test tube (or curcible) place about one spatula of  $\text{MnO}_2$  (or  $\text{Fe}_2\text{O}_3$ ). Heat the test tube until all the moisture of the catalyst is taken off but do not apply the flame continuously to one spot, otherwise the test tube may begin to melt. Allow the test tube to cool to room temperature and then weigh it (up to two decimal points) as  $W_0$ . Add 2 to 4 grams of  $\text{KClO}_3$  mix well and weigh again as  $W_1$ . Calculate the weight of  $\text{KClO}_3$  as  $W_2$ .

Clamp the test tube in a diagonal position and heat first gently and then more strongly. The mixture will first melt and then effervesce (produce gas) strongly and finally solidify into a puffy mass. Heat the entire test tube to redness; maintain the temperature for 15 minutes. Be sure not to apply the flame too long to one spot, or the glass may melt.

Cool the test tube slowly by gradually closing the air events on the Bunsen burner until the flame is brightly luminous. Shut off the burner, allow the test tube cool completely and weigh it, record the weighing as  $W_3$ . Heat the test tube and its contents to redness for another 15 min., cool and reweigh as  $W_4$ . If the values of  $W_3$  and  $W_4$  agree, you may assume that all the oxygen has driven off. If not, you must heat and reweigh until two consecutive weighing give the same value. List the weighing results below  $W_4$  on report sheet. Your last weighing is  $W_f$ , obviously  $W_f$  must be the same as  $W_4$ .

Calculate the weight of oxygen given off, report as  $W_{\text{ox}}$ .

Calculate percent oxygen by weight in  $\text{KClO}_3$ . This is experimental %.

Calculate the theoretical % of oxygen in  $\text{KClO}_3$  (At.Wt (g/mol) O= 16; Cl= 35.5; K= 39.1)

Calculate % error.

## Report Sheet A

Student's Name:

Lab. Instructor name & sign. :

Lab. Section :

Date :

---

### Name of the Experiment:

Weigh of the test tube and the catalyst,

$W_0 =$

Weigh of the test tube, catalyst and  $\text{KClO}_3$ ,

$W_1 =$

Weight of  $\text{KClO}_3$

$W_2 = W_1 - W_0 =$

Weight of the test tube and the contents after first heating,

$W_3 =$

Weight of the test tube and the contents after second heating,

$W_4 =$

Weight of the test tube and the contents after last heating,

$W_f =$

Weight of oxygen given off,

$W_{ox} = W_1 - W_f =$

Experimental % of oxygen in  $\text{KClO}_3$ ,

$\%O = (W_{ox}/W_2) \cdot 100 =$

Theoretical % of oxygen in  $\text{KClO}_3$  by weight

$=$

Percent error,

$\%E =$

**Report Sheet B** *(to be submitted to the instructor)*

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

Weight of the test tube and the catalyst,

$W_0 =$

Weight of the test tube, catalyst and  $\text{KClO}_3$ ,

$W_1 =$

Weight of  $\text{KClO}_3$

$W_2 = W_1 - W_0 =$

Weight of the test tube and the contents after first heating,

$W_3 =$

Weight of the test tube and the contents after second heating,

$W_4 =$

Weight of the test tube and the contents after last heating,

$W_f =$

Weight of oxygen given off,

$W_{ox} = W_1 - W_f =$

Experimental % of oxygen in  $\text{KClO}_3$ ,

$\%O = (W_{ox}/W_2) \cdot 100 =$

Theoretical % of oxygen in  $\text{KClO}_3$  by weight

$=$

Percent error,

$\%E =$

## EXPERIMENT NO: 4      FORMULA AND COMPOSITION OF A HYDRATE

### Chemicals and Apparatus

\*Copper Sulphate,  $\text{CuSO}_4$

\*Test Tube, Crucible or Beaker

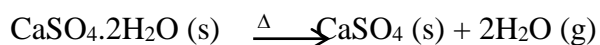
\*Bunsen Burner or Hotplate

### PURPOSE

The aim of this experiment is to determine the percentage of water in a hydrated compound and to find the formula of the hydrate.

### THEORY

Hydrates are crystalline compounds in which one or more molecules of water are combined with each formula unit of salt. This water of hydration is not bounded tightly into the crystalline structure and can be driven off by heating a sample of a hydrate. If the hydrate is colored, a color change usually results upon heating. Each hydrate salt has a fixed number of water molecules associated with it, called waters of hydration or water of crystallization. When a salt holds waters of hydration, we call it a hydrated salt. The compound without the water is said to be anhydrous.



Some anhydrous salts can absorb moisture from the air to become hydrated. These salts are said to be hygroscopic. Some hygroscopic salts are useful as drying agents. A common example is silica gel, made from sodium silicate,  $\text{Na}_2\text{SiO}_3$ , which is usually packaged with optical or electronic devices shipped by boat. There are some hygroscopic compounds that can absorb so much moisture from their surroundings that they eventually dissolve in the absorbed water. An example of this type of compound is calcium chloride,  $\text{CaCl}_2$ . This type of compound is said to be deliquescent.



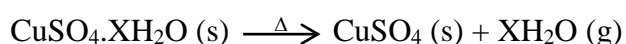
## PROCEDURE

### Finding the hydrated formula of $\text{CuSO}_4 \cdot \text{XH}_2\text{O}$

Place a crucible and cover on a clay triangle, supported on an iron ring (If necessary, clean and dry the crucible and cover.). Heat the crucible gently for about one minute, then heat strongly for about 5 minutes. Allow the crucible and cover to cool. Handle the hot or cooled crucible and cover using your crucible tongs. Weigh the cooled crucible and cover and record the mass to 0.001 g. Weigh the cover of the crucible separately, and record its mass. (This second step will allow you to still determine your final results if the crucible cover falls off the crucible and breaks during the experiment.)

Weigh 1.00 to 1.500 g of  $\text{CuSO}_4 \cdot \text{XH}_2\text{O}$ . Place the hydrate into the crucible and weigh the crucible, cover, and hydrate together. Record the mass to 0.001 g. Place the crucible and its contents on a clay triangle. Adjust the lid on the crucible so it is slightly ajar. Heat gently with a Bunsen burner for one minute. Continue to heat with a moderate flame for 5 minutes. Increase the heat to high and heat for an additional 5 minutes - the bottom of the crucible will glow with a red color. Allow the crucible to cool. When the crucible is cool, close the lid and weigh the crucible, cover, and its contents. Record the mass to 0.001 g. Save the crucible and contents until you have completed your calculations for the experiment.

After completing the calculations, inspect the compound inside the crucible. Use your crucible tongs or a glass stirring rod to press down on the compound.



*Blue*

*White*

This process is reversible. By adding water to the anhydrous salt, the hydrate will reform.

## CALCULATIONS

Calculating Experimental percentage and water of crystallization

### I. Experimental percentage:

The experimental percentage of water in a hydrate is found by comparing the mass of water driven off to the total mass of the compound, expressed as a percentage.

The mass of water lost is found by difference:

$$\text{Hydrate (grams)} - \text{anhydrous salt (grams)} = \text{water (grams)}$$

The experimental percentage of water is

$$\frac{\text{mass of water}}{\text{mass of hydrate}} \times 100 = \% \text{ of water}$$

### II. Water of crystallization:

**Example :** Calculate the water of crystallization for an unknown hydrate that is found to contain 30.6 % water. The formula mass of the anhydrous salt (AS) is 245 g/mole.

**Solution:** The unknown hydrate is 30.6 % water. Subtracting from 100 %, the hydrate must be 69.4 % anhydrous salt. If we make the sample 100 g, the mass of water is 30.6 g and the anhydrous salt 69.4 g. to calculate the moles of water and the anhydrous salt (AS)

$$30.6 \text{ g H}_2\text{O} \times \frac{1 \text{ mole H}_2\text{O}}{18.0 \text{ g H}_2\text{O}} = 1.70 \text{ moles H}_2\text{O}$$

$$69.4 \text{ g AS} \times \frac{1 \text{ mole AS}}{245 \text{ g AS}} = 0.283 \text{ mole AS}$$

To find the water of crystallization, simply divide the mole ratio of water to anhydrous salt.

$$\frac{1.70 \text{ moles H}_2\text{O}}{0.283 \text{ mole AS}} = 6.01 \approx 6$$

The water of crystallization is always a whole number; therefore the formula for the unknown hydrate is  $\text{AS} \cdot 6\text{H}_2\text{O}$ .

## Report Sheet A

Student's Name:

Lab. Instructor name & sign. :

Lab. Section :

Date :

---

**Name of the Experiment:**

**Calculate and write down the hydrated formula of  $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$**

(1) Mass of crucible and lid .....g

(2) Mass of crucible, lid and hydrate .....g

(3) Mass of hydrate (2) – (1) .....g

(4) Mass of crucible, lid and anhydrous salt (after heating) .....g

(5) Mass of water .....g

(6) Percentage of water .....%

Calculations:

Unknown Number: .....

Formula of hydrate: .....

**Report Sheet B** (*to be submitted to the instructor*)

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

Calculate and write down the hydrated formula of  $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$

(1) Mass of crucible and lid .....g

(2) Mass of crucible, lid and hydrate .....g

(3) Mass of hydrate (2) – (1) .....g

(4) Mass of crucible, lid and anhydrous salt (after heating) .....g

(5) Mass of water .....g

(6) Percentage of water .....%

Calculations:

Unknown Number: .....

Formula of hydrate: .....

## **EXPERIMENT NO: 5      DETERMINATION OF HEAT OF FUSION**

### **Chemicals and Apparatus**

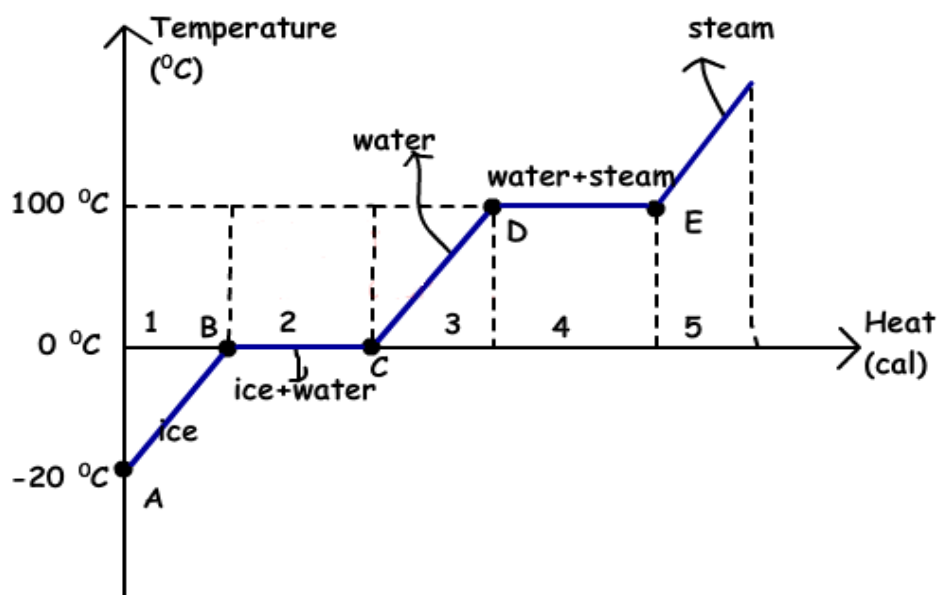
- \*Ice
- \*Beakers
- \*Thermometer
- \*Bunsen Burner or Hotplate
- \*Clamp and rings

### **PURPOSE**

In this section the amount of heat required to melt a definite amount of a solid will be determined. The solid you will use is ordinary ice. A weighed quantity of water in a beaker will be heated to a definite and measured temperature. In to this warm water, a quantity of ice will be introduced. The ice will melt, the melted ice will mix with the water already there, and all will come to the same temperature. This temperature will be measured.

### **THEORY**

The energy required to change a gram of a substance from the solid to the liquid state without changing its temperature is commonly called its "heat of fusion". This energy breaks down the solid bonds, but leaves a significant amount of energy associated with the intermolecular forces of the liquid state.



**Figure 2.** Phase Change Diagram for Water

Heat is thermal energy which "flows" from a hotter object to a cooler one because of the temperature difference between them. Heat flows through conduction, convection, radiation, or some combination of these processes. In mechanical or electrical measurements energy is usually measured in Joules, but in thermal measurements the "calorie" is usually used as the energy unit. One calorie is equal to 4.186 Joules. Heat is usually represented as  $Q$ . If two objects at different temperatures are brought together heat will flow from the warmer to the cooler object until the temperatures are equal. This process is called heat exchange. The heat lost by the warmer object must equal the heat gained by the cooler one

$$Q_{lost} = Q_{gained}$$

This can be used to find another property of a substance called specific heat. Specific heat,  $c$ , is defined as the amount of heat required to change the temperature of one kilogram of a substance  $1\text{ }^{\circ}\text{C}$ , the unit for specific heat is  $\text{J/kg}^{\circ}\text{C}$ , Joule per kilogram - degree. The equation for heat can then be rewritten:

$$Q = m.c.\Delta T$$

The calorie (cal) is defined as the amount of thermal energy required to raise the temperature of one gram of pure water from 14.5 °C to 15.5°C. Thus the specific heat of water in this temperature range is 1.00 cal/g°C. The "dietary calorie" or "Calorie" (spelled with a capital "C") which is used to define the energy equivalent of foods, is equal to a kilocalorie.

$$1 \text{ kcal} = 1 \text{ Cal} = 10^3 \text{ cal} = 4186 \text{ J}$$

When a substance is undergoing a phase transition, that is, changing from one physical state to another, it will gain or lose heat without a change in temperature until the phase transition is complete. The amount of heat necessary to melt a substance is called the latent heat of fusion,  $L$ , and has units of J/kg. The amount of heat necessary to completely change a substance from solid to liquid, without changing its temperature is given by

$$Q = m.L \text{ and } L = H/M \text{ (M: Atomic or Molecular weight)}$$

In this experiment you will find the latent heat of fusion of ice. When ice at 0 °C is mixed with warm water at some initial temperature,  $T_i$ , the water will cool to a final temperature,  $T_f$ . In the process it will lose heat equal to  $m_{\text{water}} \cdot c_{\text{water}} (T_i - T_f)$ . Some of the lost heat will be used to melt the ice,  $m_{\text{ice}} \cdot L_f$ , and the rest will raise the temperature of the melted ice to the final temperature of the water. Therefore,

$$m_{\text{water}} \cdot c_{\text{water}} (T_i - T_f) = m_{\text{ice}} L_f + m_{\text{ice}} \cdot c_{\text{water}} (T_f - 0^\circ\text{C}).$$

The amount of heat required to raise the temperature of one mole (abbreviated mol) of a substance 1 °C is called its molar specific heat.

A mole of any substance contains  $6.022 \times 10^{23}$  particles and has a mass equal to the gram molecular mass of the substance. Thus one mole of diatomic oxygen has a mass of 32.0 g. In the middle of the nineteenth century, Dulong and Petit found that the molar specific heats of almost all solids have the same value when measured at around room temperature. That value is approximately 25 J/mol °C. In 1905, Einstein developed a crude theoretical model, based on quantum ideas, which predicted this result and also explained why these values change when measurements are made at lower temperatures. In fact, for all substances, the molar specific heat approaches zero as the temperature approaches absolute zero.

## PROCEDURE

Clean and dry your 400 cc (mL) beaker, then weigh to the nearest gram on the balance. Half fill it with water and weigh it again. Arrange a ring and a ring stand to heat this water and warm it to about 50 °C. Meantime obtain a sample of crushed ice about enough to half fill your 250 mL beaker. Dry the ice as well as you can by rubbing it in a clean, lint-free towel. Measure the temperature of the water in the beaker, recording this temperature as “Initial Temperature”. Quickly dump your ice into it. Stir rapidly with the thermometer and as soon as the last sliver of ice has disappeared, read the thermometer again. Record this temperature as “final temperature”. Weigh the beaker again in order to tell how much ice was added and record the weight.

Compute the heat of fusion of ice as indicated on the theory section; The water in the beaker at the end of the experiment came from two sources: the water originally there and the water produced by the melted ice. When the water cooled from its initial temperature of about 50 °C to the final temperature, it lost an amount of heat. Calculate this heat loss for the water. Calculate the heat gained by the ice-water.

Calculate the amount of heat required to warm the amount of ice water you had from its initial temperature of 0 °C to its final temperature. Observe that this amount of heat is far less than the total amount of heat gained by the ice-water. The difference is the amount of heat required to melt the ice without raising its temperature. Compute this difference. This amount of heat was required to melt whatever weight of ice you used. Noting that the weight of ice you used is the same as the weight of ice-water is produced on melting, compute the heat required to melt just 1 g. of ice. This is the heat of fusion. Taking the molar weight of water as 18 g per mole, compute also the molar heat of fusion of water.

Melting Point (1 atm. pressure) : 0 °C

Specific Melting Heat of Ice (L) : 80 cal/g

Molar Heat Melting of Ice (H) : 1440 cal/g

Specific Heat of Water ( $c_{\text{water}}$ ) : 1 cal/g °C

Specific Heat of Ice ( $c_{\text{ice}}$ ) : 0,5 cal/g °C

Specific Heat of Glass ( $c_{\text{glass}}$ ) : 0,12 cal/g °C



## Report Sheet A

Student's Name:

Lab. Instructor name & sign. :

Lab. Section :

Date :

---

Name of the Experiment:

1. Calculate the Specific Melting Heat of Ice (L) and compare with the theoretical value

Mass of water in beaker : .....g

Initial temperature of water in beaker : ..... °C

Mass of beaker : .....g

Initial temperature of beaker : ..... °C

Mass of the ice : .....g

Initial temperature of ice : ..... °C

Final temperature of (beaker + water + ice) : ..... °C

2. Calculate the Molar Heat Melting of Ice (H) and compare with the theoretical value

### Question

Calculate the heat required to melt 25 g of ice? (Initial temperature: -10 °C)

**Report Sheet B** *(to be submitted to the instructor)*

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

- 1. Calculate the Specific Melting Heat of Ice (L) and compare with the theoretical value**

Mass of water in beaker : .....g

Initial temperature of water in beaker : ..... °C

Mass of beaker : .....g

Initial temperature of beaker : ..... °C

Mass of the ice : .....g

Initial temperature of ice : ..... °C

Final temperature of (beaker + water + ice) : ..... °C

- 2. Calculate the Molar Heat Melting of Ice (H) and compare with the theoretical value**

**Question**

Calculate the heat required to melt 25 g of ice? (Initial temperature: -10 °C)

## EXPERIMENT NO: 6      PREPARATION OF $K_3[Fe(C_2O_4)_3].3H_2O$ and % YIELD

### Chemicals and Apparatus

\*Potassium Oxalate monohydrate,  $K_2C_2O_4.H_2O$

\*Ferric Chloride (Iron(III)Chloride) hexahydrate,  $FeCl_3.6H_2O$

\*Beaker

\*Filter Paper

\*Funnel

### PURPOSE

In this experiment, the student will prepare inorganic compound Potassium trioxalatoferrate(III)trihydrate ( $K_3[Fe(C_2O_4)_3].3H_2O$ ), determine the limiting reactant and calculate the percentage yield (% yield) of the reaction.

### THEORY

Potassium trioxalatoferrate(III)trihydrate is a green crystalline salt, soluble in hot water but rather insoluble in cold water. It can be prepared by the reaction of  $K_2C_2O_4.H_2O$  with  $FeCl_3.6H_2O$ .

In chemistry, yield, also referred to as reaction yield, is the amount of product obtained in a chemical reaction. The absolute yield can be given as the weight in grams or in moles (molar yield). The percentage yield, which serves to measure the effectiveness of a synthetic procedure, is calculated by dividing the amount of the obtained product (actual yield) by the theoretical yield. The amount of the product calculated from the balanced equation of the reaction is the **theoretical yield** of that reaction. Most reactions, however, do not go to completion. In practice we always get less. The amount of the product produced at the end of the reaction is the **actual yield**.

$$\text{Percentage Yield (\% Yield)} = \frac{\text{Actual Yield}}{\text{Theoretical yield}} \cdot 100$$

The reactant which is completely used up in the reaction is called **limiting reactant**, the one which remains is called the **excess reactant**. The theoretical yield is therefore calculated based on the molar amount of the limiting reactant, taking into account the stoichiometry of the reaction. For the calculation it is usually assumed that there is only one reaction involved. The ideal or theoretical yield of a chemical reaction would be 100%, a value that is impossible to achieve due to limitations in measurement accuracy. Yields may appear to be above 100% when products are impure. Purification steps always lower the yield and the reported yields usually refer to the yield of the final purified product.

## PROCEDURE

Weigh approximately 4.5 g of Potassium Oxalate monohydrate,  $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$  into a 250 mL beaker. Add 30 mL of distilled water and heat to dissolve (do not boil). In a second small beaker dissolve 4.4 g of Ferric Chloride (Iron(III)Chloride) hexahydrate,  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  in a minimum amount of cold water (10-15 mL). Add  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  solution to the warm oxalate solution, stir with a glass rod. Allow the product to crystallize (away from strong sunlight) by cooling the solution in an ice-water mixture. Filter off the cold mixture using a funnel and filter paper and transfer the filter paper with precipitate on a watch glass and place it in desiccator to dry. The product is  $\text{K}_3[\text{Fe}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$ .

## Report Sheet A

Student's Name:

Lab. Instructor name & sign. :

Lab. Section :

Date :

---

Name of the Experiment:

1. Write down and balance the complete reaction



2. Determine the limiting reactant and calculate the percentage yield

**Report Sheet B** (*to be submitted to the instructor*)

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

- 1. Write down and balance the complete reaction**



- 2. Determine the limiting reactant and calculate the percentage yield**

## **EXPERIMENT NO: 7      EQUIVALENT WEIGHT OF A METAL**

### **Chemicals and Apparatus**

- \*Metal Ribbon
- \* Hydrochloric Acid, HCl
- \*Burette
- \*Beaker
- \*Ruler

### **PURPOSE**

In this experiment, the student will measure the equivalent weight of a metal by means of its reaction with an acid.

### **THEORY**

The equivalent weight of a substance (ew) can be described in a number of ways, depending on what type of reaction is involved. All of these, however, are simply various ways of looking at what happens to electrons. Equivalent weight (ew) is the number of grams of a substance which will gain or lose one mole of electrons.

By that definition, then, the equivalent weight of any metal is the number of grams of metal which loses one mole of electrons. Since we can not directly measure the amount of these electrons, we do it indirectly by what they do. In an acidic solution, the electrons given up by a metal are gained by hydrogen ions ( $H^+$ ), converting them to hydrogen atoms (H). The hydrogen atoms then combine to form hydrogen molecules ( $H_2$ ). We can measure the volume of this gas. A mole of electrons lost by one equivalent weight of a metal will be gained by a mole of hydrogen ions to form one-half mole of hydrogen molecules. According to Avogadro's Theory, one mole of a gas occupies 22.4 liters (22,400 ml) at standard temperature ( $273\text{ K} = 0^\circ\text{C}+273$ ) and standard pressure ( $1\text{ atm} = 760\text{ mm Hg}$ ). So one-half

mole would occupy 11,200 ml at STP. In other words, one equivalent weight of a metal would form 11,200 ml of hydrogen gas at STP.

The equivalent weight of any metal can then be calculated based on the volume of hydrogen produced by a given amount of metal and the above proportion. You will measure the equivalent weight of a metal by means of its reaction with an aqueous solution of hydrochloric acid. Since the gas is collected over water, you will have to correct the measured volume by subtracting the vapor pressure of water. Look up the vapor pressure of water at room temperature in the table at the end of this exercise. Because the reaction and collection of hydrogen are not done at standard temperature and pressure (STP), you will also have to convert the measured volume of hydrogen mathematically to what it would be at STP.

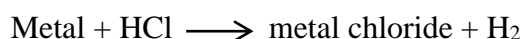


## PROCEDURE

Cut off about 7 cm of the metal ribbon which will be obtained from your instructor, weigh the ribbon and record it. Wad the ribbon into a small ball and form a small cage around it with a piece of copper wire. Leave about 5 cm of the copper wire straight to serve as a handle. Pour 10 ml of 12 M HCl into the gas collecting tube. Then carefully pour enough water into the gas collecting tube to fill it to the brim – tilt the tube slightly and pour the water slowly down the inner wall so that the acid at the bottom is disturbed as little as possible.

Place the caged metal into the water about 3 or 4 cm and hold it in place with a one-hole rubber stopper (which holds the handle of the cage between the glass the and rubber). DON'T PUT YOUR FINGER OVER THE HOLE IN THE STOPPER AS YOU PRESS IT FIRMLY INTO PLACE! Make sure that no air is trapped in the tube and that the hole of the stopper is full of water.

Place about 300 ml of tap water in a 600 ml or larger beaker. Cover the hole in the stopper with your finger, carefully turn the tube bottom-up, and insert the stoppered end into the water in the beaker. When the end is under the water, remove your finger. Hold the collecting tube vertically in this position with a buret clamp on a ring stand. The HCl, now at the top, is heavier than water and will sink towards the caged metal. When it reaches the metal, the following reaction occurs:



When the reaction is complete (the metal is gone and no more bubbles form), let the apparatus cool for 10 minutes to reach room temperature.

Measure and record the room temperature, and convert it from Celsius to Kelvin ( $K = ^\circ\text{C} + 273$ ).

Write down the current air pressure, mm Hg (provided by the instructor).

Record the vapor pressure of water at room temperature when you look it up.

Measure the volume of hydrogen produced by the reaction by reading the graduations on the gas collecting tube, which is marked in ml ( $V_H$ ).

The pressure of the  $\text{H}_2$  inside the collecting tube ( $P_H$ ) can be calculated by the formula:

$$P_H = P_A - P_V - P_W$$

$P_A$  is atmospheric pressure.  $P_V$  is the vapor pressure of water at room temperature.  $P_W$  is the pressure due to the column of water inside the tube.

To determine  $P_W$ , measure how far the water comes up the tube above the surface of

the water in the beaker. Measure this in mm with a ruler or meter stick.

Divide this height in mm by 13.6 (the density of mercury) to get the pressure due to the column of water ( $P_w$ ).

Now you can calculate the pressure of  $H_2$  in the tube ( $P_H$ ).

Use the following equation to calculate the volume of hydrogen at room temperature and pressure to the volume at STP:

$$V_S = (V_H \times P_H \times T_S) / (P_S \times T_R)$$

$T_S$  is standard temperature, 273 K and  $P_S$  is standard pressure, 760 mm Hg.

The experimental equivalent weight of the metal can then be calculated by the following equation:

$$ew = (g \text{ metal} \times 11200 \text{ ml}) / V_S$$

## Report Sheet A

Student's Name:

Lab. Instructor name & sign. :

Lab. Section :

Date :

---

Name of the Experiment:

Calculate the equivalent weight of metal (ew).

Weight of metal ribbon .....g

Temperature ..... $^{\circ}\text{C}$

Temperature ( $T_R$ ) .....K

$P_A$  .....mmHg

$P_V$  (vapor pressure) .....mmHg

$V_H$  .....mL

$P_W$  .....mm

$P_W$  .....mmHg

$P_H$ .....mmHg

$V_S$  :

ew:

**Report Sheet B** (*to be submitted to the instructor*)

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

**Calculate the equivalent weight of metal (ew).**

Weight of metal ribbon .....g

Temperature .....<sup>0</sup>C

Temperature (T<sub>R</sub>) .....K

P<sub>A</sub> .....mmHg

P<sub>V</sub> (vapor pressure) .....mmHg

V<sub>H</sub> .....mL

P<sub>W</sub> .....mm

P<sub>W</sub> .....mmHg

P<sub>H</sub>.....mmHg

V<sub>S</sub> :

ew:

## EXPERIMENT NO: 8      OXIDATION-REDUCTION REACTIONS

### Chemicals and Apparatus

\*Copper (II) Sulfate pentahydrate,  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$

\*Silver Nitrate,  $\text{AgNO}_3$

\*Nitric Acid,  $\text{HNO}_3$

\*Potassium Iodide,  $\text{KI}$

\*Sodium Nitrite,  $\text{NaNO}_2$

\*Metallic Zinc

\*Metallic Copper

### PURPOSE

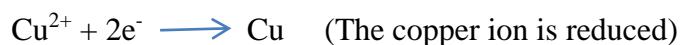
The aim of this experiment is to illustrate the electron exchange of metals in different solutions.

### THEORY

**Oxidation** is the loss of electrons or an increase in oxidation state by a molecule, atom, or ion. **Reduction** is the gain of electrons or a decrease in oxidation state by a molecule, atom, or ion.

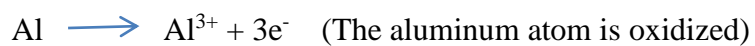
An oxidation-reduction reaction is a chemical reaction in which electrons are transferred from one element to another element. The element that gains the electrons has a decrease in its oxidation number (because electrons have a negative charge). Reduction is the part of the oxidation-reduction reaction in which an element's oxidation number is reduced.

For example when a cupric ( $\text{Cu}^{2+}$ ) ion gains two electrons to form a copper atom, the copper ion is being reduced because its oxidation number is being reduced from 2+ to zero.



The element that loses electrons has an increase in its oxidation number. Oxidation is the part of an oxidation-reduction reaction in which an element's oxidation number is increased by losing electrons.

For example when an aluminum atom loses three electrons to form an aluminum ion, the aluminum atom is being oxidized.



In any oxidation-reduction reaction the number of electrons gained by the element that is reduced must equal the number of lost by the element that is oxidized.

## PROCEDURE

1. Take 2 mL of  $\text{AgNO}_3$  solution into a test tube. Into this solution put a small copper ribbon (~0.2 g). Record what you observe in 5 or 10 minutes.
2. Put a small copper ribbon into a test tube and add 3 mL of concentrated Nitric acid. Wait for approximately 10 minutes and record your observations.
3. Boil some water in a 400 mL beaker and place the tube in step 2 into this beaker. Wait until no gas evolves from the tube and record your observations.
4. Put a metallic zinc piece (~0.2 g) into the test tube of step 3 and write your observations in 10 minutes.
5. Take 2 mL of copper sulfate solution into a test tube and insert a small piece of metallic zinc. Record your observations.
6. Put about 0.1 g of KI into a test tube, add 10 mL of distilled water, shake for 2-3 minutes and put 0.1 g of  $\text{NaNO}_2$  into this solution. Write the changings that you observe.

## Report Sheet A

Student's Name:

Lab. Instructor name & sign. :

Lab. Section :

Date :

---

Name of the Experiment:

**1. Step 1,**

- Write the two half reactions and balance them
- Where does the blue color of the solution come from?

**2. Step 2,**

- Write down the reaction and indicate the gas evolving

**3. Step 3,**

- Write down the half and overall reactions

**4. Step 5,**

- Write the half reactions and balance them
- What can you say about the functions of  $\text{SO}_4^{2-}$  and  $\text{NO}_3^-$  ions in these reactions?



**Report Sheet B** *(to be submitted to the instructor)*

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

**1. Step 1,**

- Write the two half reactions and balance them

- Where does the blue color of the solution come from?

**2. Step 2,**

- Write down the reaction and indicate the gas evolving

**3. Step 3,**

- Write down the half and overall reactions

**4. Step 5,**

- Write the half reactions and balance them
  
- What can you say about the functions of  $\text{SO}_4^{2-}$  and  $\text{NO}_3^-$  ions in these reactions?

## EXPERIMENT NO: 9      SOAP

### Chemicals and Apparatus

- \*Sunflower Oil or Olive Oil
- \*Sodium Hydroxide, NaOH
- \*Ethanol, C<sub>2</sub>H<sub>5</sub>OH
- \*Sodium Chloride, NaCl
- \*Beakers

### PURPOSE

The aim of this experiment is to synthesize soap and to understand why soap is useful in cleaning and understand the chemistry behind the making of soap.

### THEORY

A triglyceride consists of three fatty carboxylic acids bonded to glycerol. The fatty acid consists of a long carbon chain terminated with a carboxylic acid group. The carboxylic acid group consists of a carbon atom bonded to two oxygen atoms. One of the bonds is a single bond and the other a double bond. Normally in the carboxylic acid group the singly bonded oxygen atom has a hydrogen atom attached to it. However, in this case it is bonded to a carbon atom, which is part of a three-carbon chain. Notice that each of these three carbon atoms has a carboxylic acid group attached to them, thus the name "tri"glyceride. Triglycerides are called fats if they are solid at room temperature and oils if they are liquid at room temperature. Triglycerides are not soluble in water as evidenced by the beading of water on lard or the separation of oil from water. When triglycerides are reacted with sodium hydroxide, the bond between the oxygen atom of the carboxylic acid group and the carbon atom of the glycerol is broken. This process is called **saponification**.

The oxygen atom picks up the sodium atom from the sodium hydroxide and thus makes this end of the fatty acid chain soluble in water. This sodium salt of a fatty acid is called soap. The hydroxide group ( $\text{OH}^-$ ) from sodium hydroxide then attaches to the glycerol molecule. When all three fatty acids are removed, the reaction is complete. The presence of ethanol during this process allows the reaction to take place at medium heat. While heating the mixture, ethanol and water will evaporate away. When the reaction is complete, the glycerol needs to be separated from the soap. This is done by adding water to the mixture to make it more liquid in texture and then by adding a saturated solution of salt water. The glycerol is more soluble in the salt water than the soap and, therefore, allows these two chemicals to be separated. This process is referred to as "salting out" the soap.

The unique properties of soap lie in the fact that one end of the chain is water soluble (hydrophilic) and the other end is not (hydrophobic). In the process of washing with soap, a particle of grease is surrounded by soap molecules. The hydrophobic ends attach to the grease particle, thus leaving the hydrophilic end exposed to the water. This allows the grease particle to freely move around in the water and, therefore, be washed away. The fats and oils used in making soap consist mainly of seven different fatty acids. If all of the carbon bonds in the fatty acid chain are single bonds, we have a saturated fatty acid. If any of the carbon bonds are double bonds, we have an unsaturated fatty acid. Unsaturated fats can be converted to saturated fats by adding hydrogen atoms at the locations of the double bonds. The amount of unsaturated fatty acids in the soap can affect the firmness of the soap.

*CAUTION: Sodium hydroxide is a strong base and corrosive. Ethyl alcohol is flammable. Wear latex gloves and goggles during the lab to protect yourself from the sodium hydroxide.*

## PROCEDURE

Place about 20 g (~25 mL) sunflower oil or olive oil in your 500 mL beaker. Add 15 mL ethanol and 15 mL of NaOH solution (dissolve 6 g NaOH in 15 mL of water). Stir the mixture constantly with a glass stirring rod and heat gently (medium heat) on a hot plate until the alcohol evaporates (~30 min.). Allow the mixture to cool and then add 100 mL of NaCl solution (dissolve 5 g NaCl in 100 mL water). Filter off the soap mixture and wash the collected soap precipitate with 15 mL of ice water. Dry your soap.

### ***How does soap work to clean things?***

*Soap is an emulsification agent. Oil and grease are not soluble in water (they don't dissolve in water), which is why, for instance, oil and water will separate when combined. This is because oils are non-polar molecules, but water is a polar molecule, and non-polar things do not mix well with polar things. In order to remove oil from clothes or from your dishes with water, the oils must be made soluble by the process of emulsification. The non-polar end adsorbs the oil or other hydrophobic dirt. The ionic end is highly soluble in water. This allows for an emulsion to be formed. The alkali metal (sodium or potassium ion) does not play a role in the action of the soap. Soap use is not a chemical reaction, but a physical one. Under normal conditions, the soap does not react with the dirt chemically. If "hard water" minerals are present (magnesium or calcium) these can chemically react with the soap and lessen its effectiveness by removing the soap from solution. The structure of the emulsion is such that the oil or oily dirt is surrounded by soap molecules with the ionic part of the molecules toward the outside where water will react with the ionic end (by hydrogen bonding) and keep the oil in "solution." Hot water helps in the formation and suspension of the emulsion. Other additives are sometimes added to soap for antibacterial or antifungal effects, but these are not the main ingredients -- they just enhance the primary function of emulsifying oils in water. Soap alone cannot remove dirt or kill germs-- it is the act of running water along with the oils in the soap that help loosen the dirt and rinse it off of your skin. Soap will not destroy the dirt or even germs but the ingredients in soaps such as lye, will make a hostile environment where germs cannot survive. Anti bacterial soaps do kill some bacteria but not all of them and it will not kill any viruses. So, the best way to keep your hands free and clear of germs is to rub your hands and the soap under water and make lots of lather, then rinse thoroughly with plenty of clean water.*

## Report Sheet A

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

1. Write down the reaction between Oil and NaOH

2. Why did you use ethanol?

3. Why did you use NaCl solution?

**Report Sheet B** (*to be submitted to the instructor*)

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

1. Write down the reaction between Oil and NaOH

2. Why did you use ethanol?

3. Why did you use NaCl solution?

## **EXPERIMENT NO: 10      BLUE BOTTLE REACTION**

### **Chemicals and Apparatus**

- \*Glucose or Dextrose
- \* Methylene blue
- \*Sodium Hydroxide, NaOH
- \*Ethanol
- \*Tap Water
- \*Flasks

### **PURPOSE**

This experiment illustrates how to perform the blue bottle chemistry demonstration, in which a blue liquid becomes clear, and then returns to blue when swirled around.

### **THEORY**

The famous blue bottle experiment a visually dramatic way to teach reduction-oxidation (redox) chemistry. Students from grade school to grad school find this reaction memorable and it is considered a classic staple in chemical demonstration shows. A half-full bottle of colorless liquid turns blue when shaken, and when the bottle is allowed to sit still, the color fades. Shaking the bottle again causes the color to reappear like magic.

On the molecular level, the blue bottle experiment is a complex system composed of ethanol, the simple sugar glucose, the dye methylene blue, the hydroxide ion, and oxygen from the atmosphere. The color change occurs do to a pair of competing reduction-oxidation reactions. Hence, the blue bottle experiment is a wonderful tool for introducing the key concepts of reduction and oxidation.

All redox reactions involve electrons being transferred from one compound, the reducing agent, to another compound, the oxidizing agent. The term "reduction" means "gain of electrons". This seems like an odd choice of terminology since "gain" and "reduce" are

usually considered antonyms. However, because the electron has an electrical charge of negative one, gaining electrons will reduce the charge of a species. The term "oxidation" means "loss of electrons" and often, but not always, involves reaction with oxygen. A common mnemonic is the phrase OIL RIG, which stands for "Oxidation Is Loss, Reduction Is Gain".

## **PROCEDURE**

Make a solution of 0.05 g of methylene blue in 50 cm<sup>3</sup> of ethanol (0.1%). Weigh 8 g of potassium hydroxide into the 1 dm<sup>3</sup> conical flask. Add 300 cm<sup>3</sup> of water and 10 g of glucose and swirl until the solids are dissolved. Add 5 cm<sup>3</sup> of the methylene blue solution. The exact quantity used is not critical. The resulting blue solution will turn colorless after about one minute. Stopper the flask.

Holding the stopper securely in place, shake the flask vigorously so that air dissolves in the solution. The color will change to blue and will fade back to colorless over about 30 seconds. The more shaking, the longer the blue color will take to fade. The process can be repeated for over 20 cycles. After some hours, the solution will turn yellow and the color changes will fail to occur.



## Report Sheet A

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

- 1. Investigate and explain how the blue bottle reaction works.**

**Report Sheet B** (*to be submitted to the instructor*)

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

- 1. Investigate and explain how the blue bottle reaction works.**

## EXPERIMENT NO: 11     SOLUTIONS

### Chemicals and Apparatus

\*Various Acids, bases and salts in the Laboratory

\*Volumetric Flasks

\*Beakers

### PURPOSE

The purpose of this experiment is to learn how to express the concentration of solutions and to prepare solutions in different concentrations.

### THEORY

The concentration of a solution

- is a macroscopic property,
- represents the amount of solute dissolved in a unit amount of solvent or of solution,
- can be expressed in a variety of ways (qualitatively and quantitatively).

A solution can be qualitatively described as;

**Dilute:** a solution that contains a small proportion of solute relative to solvent, or

**Concentrated:** a solution that contains a large proportion of solute relative to solvent.

A solution can be semi-quantitatively described as;

**Unsaturated:** a solution in which more solute will dissolve, or

**Saturated:** a solution in which no more solute will dissolve.

The solubility of a solute is the amount of solute that will dissolve in a given amount of solvent to produce a saturated solution. For example, at 0°C, we can dissolve a maximum of 35.7 g of solid NaCl in 100 mL of water (a saturated solution). Any additional solid NaCl that we add to the saturated solution simply falls to the bottom of the container and does not dissolve. There are a number of ways to express the relative amounts of solute and solvent in a solution. Which one we choose to use often depends on convenience. For example, it is sometimes easier to measure the volume of a solution rather than the mass of the solution.

## 1. Molarity

Molarity tells us the number of moles of solute in exactly one liter of a solution. (Note that molarity is spelled with an "r" and is represented by a capital M.)

We need two pieces of information to calculate the molarity of a solute in a solution:

- the moles of solute present in the solution.
- the volume of solution (in liters) containing the solute.

To calculate molarity we use the equation:

$$\text{Molarity} = \frac{\text{moles of solute}}{\text{volume of solution in liters}} \quad \text{or} \quad M = n/V$$

## 2. Normality

Normality tells us the gram-equivalent of solute in exactly one liter of a solution. (Note that normality is represented by a capital N.)

We need two pieces of information to calculate the normality of a solute in a solution:

- the equivalent weight of solute present in the solution.
- the gram-equivalent of solute present in the solution.

To calculate equivalent weight, gram-equivalent and normality we use the equations:

$$\text{Equivalent weight} = \text{molar mass} / (\text{equivalence factor}, f_{\text{eq}})$$

$$\text{Gram-equivalent} = \text{mass of compound} / \text{Equivalent weight}$$

$$\text{Normality} = \text{Gram-equivalent} / \text{Volume of solution (L)}$$

Also there is a relationship between normality and molarity;

Normality = Molarity x equivalence factor

**\*Ask your instructor about  $f_{eq}$  (equivalence factor).**

### 3. Molality

Molality,  $m$ , tells us the number of moles of solute dissolved in exactly one kilogram of solvent. (Note that molality is spelled with two "l"s and represented by a lower case  $m$ .)

We need two pieces of information to calculate the molality of a solute in a solution:

- the moles of solute present in the solution.
- the mass of solvent (in kilograms) in the solution.

To calculate molality we use the equation:

$$\text{Molality} = \frac{\text{moles of solute}}{\text{mass of solvent in kilograms}}$$

### 4. Percent Composition (by mass)

We can consider percent by mass (or weight percent, as it is sometimes called) in two ways:

- the parts of solute per 100 parts of solution.
- the fraction of a solute in a solution multiplied by 100.

We need two pieces of information to calculate the percent by mass of a solute in a solution:

- the mass of the solute in the solution.
- the mass of the solution.

Use the following equation to calculate percent by mass:

$$\text{Percent by mass} = \frac{\text{mass of solute}}{\text{mass of solution}} \times 100$$

## 5. Mole Fraction

The mole fraction,  $X$ , of a component in a solution is the ratio of the number of moles of that component to the total number of moles of all components in the solution. To calculate mole fraction, we need to know the number of moles of each component present in the solution.

The mole fraction of A,  $X_A$ , in a solution consisting of A, B, C, ... is calculated using the equation:

$$X_A = \frac{\text{moles of A}}{\text{moles of A} + \text{moles of B} + \text{moles of C} + \dots}$$

To calculate the mole fraction of B,  $X_B$ , use:

$$X_B = \frac{\text{moles of B}}{\text{moles of A} + \text{moles of B} + \text{moles of C} + \dots}$$

## PROCEDURE

Prepare the following solutions:

1. 0.1 M NaCl (250 mL)
2. 0.2 N CuSO<sub>4</sub> (500 mL)
3. 0.25 N H<sub>2</sub>SO<sub>4</sub> (100 mL) from %98 solution of H<sub>2</sub>SO<sub>4</sub> (d=1.84 g/mL)
4. % 3 solution of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>
5. % 8 solution of AgNO<sub>3</sub> from %12 solution of AgNO<sub>3</sub>

## Report Sheet A

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

**Calculate the amounts needed for the solutions in the procedure**

**1.**

**2.**

**3.**

**4.**

**5.**

**Report Sheet B** (*to be submitted to the instructor*)

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

**Calculate the amounts needed for the solutions in the procedure**

**1.**

**2.**

**3.**

**4.**

**5.**



## **EXPERIMENT NO: 12      EFFECT OF CONCENTRATION ON REACTION RATE**

### **Chemicals and Apparatus**

\*Sodium Thiosulfate,  $\text{Na}_2\text{S}_2\text{O}_3$

\*Hydrochloric Acid,  $\text{HCl}$

\*Flasks

\*Measuring cylinder

\*Chronometer

### **PURPOSE**

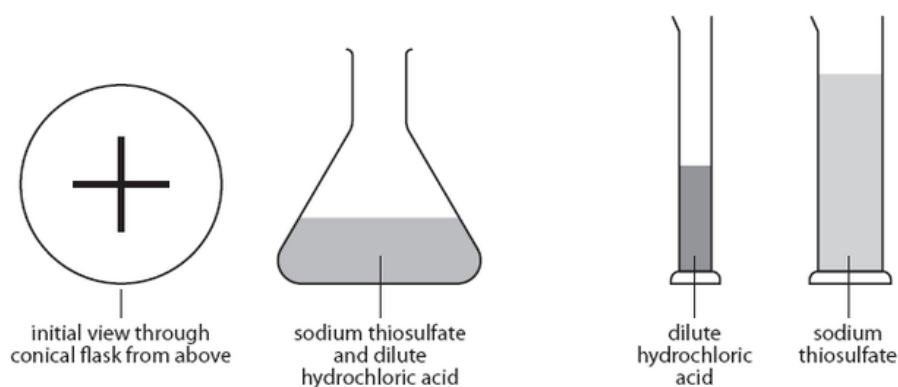
In this experiment the student will study the effect of concentration on reaction rate.

### **THEORY**

Reaction rates can be increased if the concentration of reactants is raised. An increase in concentration produces more collisions. The chance of an effective collision goes up with the increase in concentration. The exact relationship between reaction rate and concentration depends on the reaction "mechanism". This is the process involving elementary reaction steps. The slowest step controls the rate. The nature of the slow step is not obvious from the balanced equation. This is because the molecules in the reaction mixture have a range of energy levels. When collisions occur, they do not always result in a reaction. If the two colliding molecules have sufficient energy they will react.

## PROCEDURE

Put 10 mL of %5 solution of sodium thiosulfate in a flask. Measure 10 mL of %2.5 solution of hydrochloric acid in a small measuring cylinder. Add the acid and 30 mL of water to the flask and immediately start the clock. Swirl the flask to mix the solutions and place it on a piece of paper marked with a cross. Look down at the cross from above. When the cross disappears stop the clock and note the time. Record this in the table. Repeat these using different concentrations of sodium thiosulfate solution. Make up 50 mL of each solution. Mix different volumes of the sodium thiosulfate and hydrochloric acid solutions with water as shown in the table. As soon as possible, pour the solution down the sink (in the fume cupboard if possible) and wash away.



<b>Flask No</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>
HCl (mL)	10	10	10	10
Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> (mL)	10	20	30	40
Water (mL)	30	20	10	0

<b>Experiment No</b>	<b>Volume of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (mL)</b>	<b>Volume of HCl (mL)</b>	<b>Volume of H<sub>2</sub>O (mL)</b>	<b>Time taken for cross to disappear (s)</b>	<b>1/Time taken (s<sup>-1</sup>)</b>
<b>1</b>					
<b>2</b>					
<b>3</b>					
<b>4</b>					

## Report Sheet A

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

Date :

**Name of the Experiment:**

4. Write down the reaction and explain the reason of the turbidity
5. For each set of results, calculate the value of  $1/\text{time}$ . (This value can be taken as a measure of the rate of reaction).
6. Plot a graph of  $1/\text{time}$  taken on the vertical (y) axis and concentration on the horizontal (x) axis.

**Report Sheet B** (*to be submitted to the instructor*)

**Student's Name:**

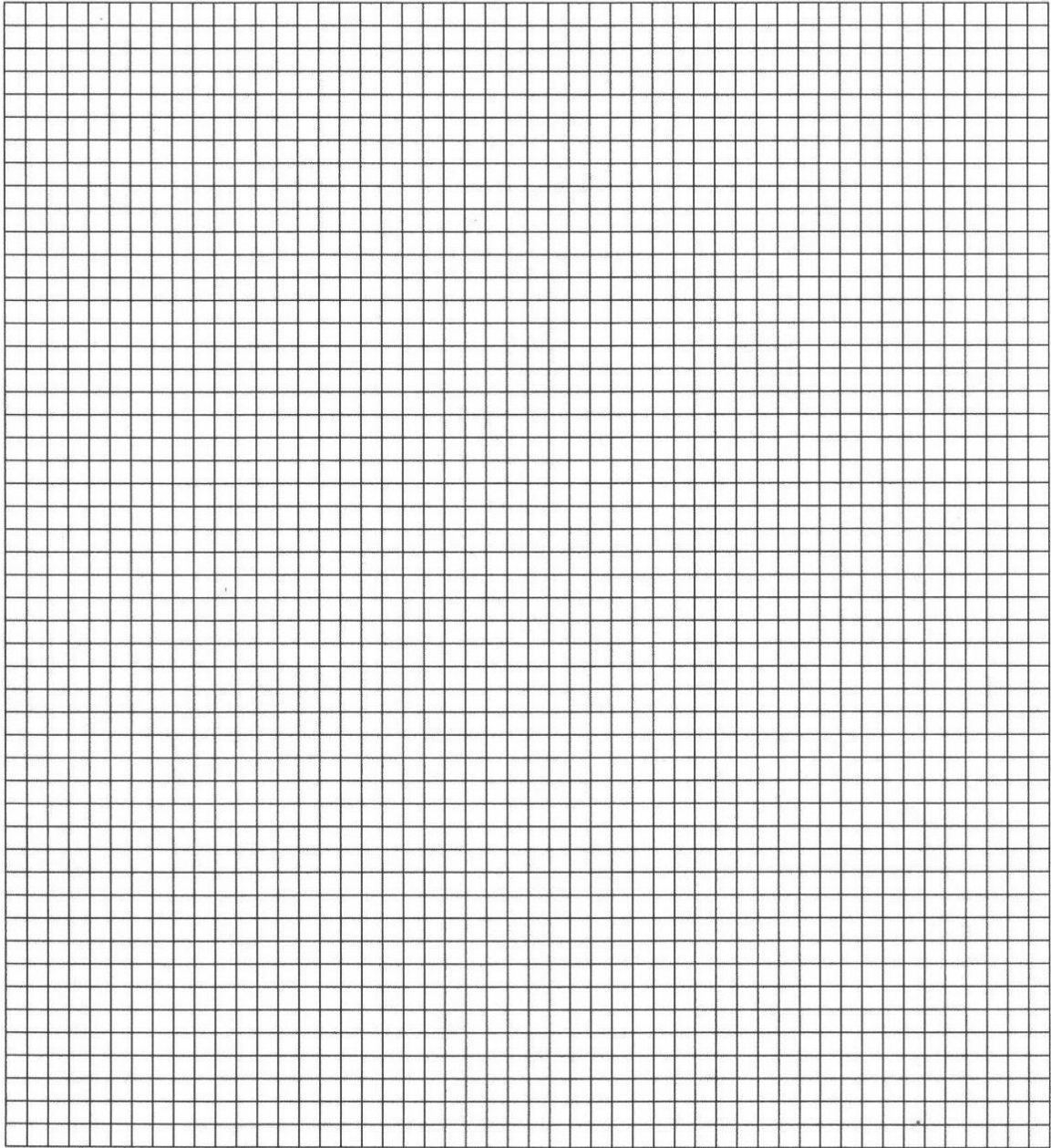
**Lab. Instructor name & sign. :**

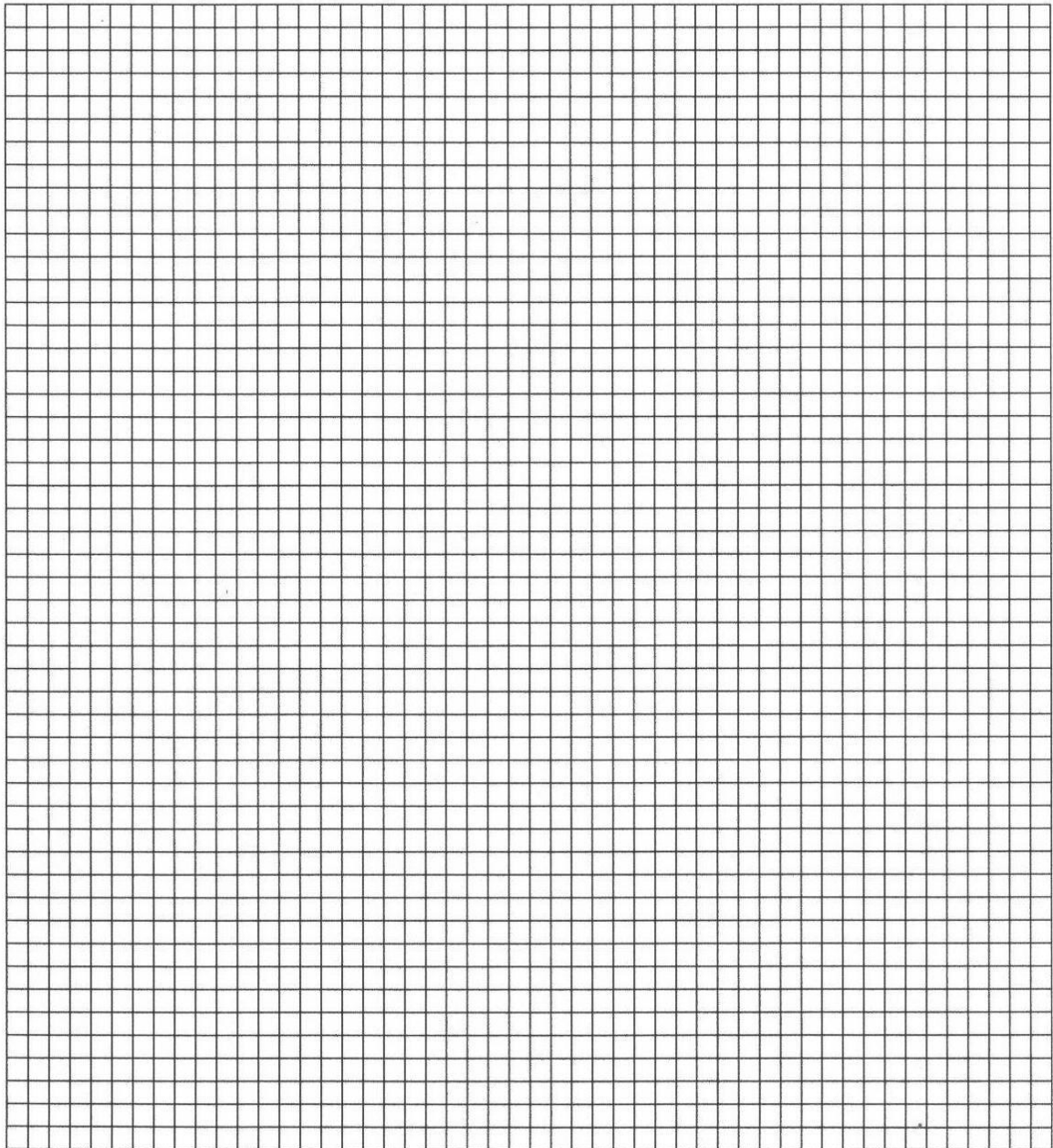
**Lab. Section :**

Date :

**Name of the Experiment:**

1. Write down the reaction and explain the reason of the turbidity
2. For each set of results, calculate the value of  $1/\text{time}$ . (This value can be taken as a measure of the rate of reaction).
3. Plot a graph of  $1/\text{time}$  taken on the vertical (y) axis and concentration on the horizontal (x) axis.





## **EXPERIMENT NO: 13      EFFECT OF TEMPERATURE ON REACTION RATE**

### **Chemicals and Apparatus**

\*Sodium Thiosulfate,  $\text{Na}_2\text{S}_2\text{O}_3$

\*Hydrochloric Acid,  $\text{HCl}$

\*Flasks

\*Measuring cylinder

\*Chronometer

\*Thermometer

### **PURPOSE**

In this experiment the student will study the effect of temperature on reaction rate.

### **THEORY**

When two chemicals react, their molecules have to collide with each other with sufficient energy for the reaction to take place. This is collision theory. The two molecules will only react if they have enough energy. By heating the mixture, you will raise the energy levels of the molecules involved in the reaction. Increasing temperature means the molecules move faster. This is kinetic theory.

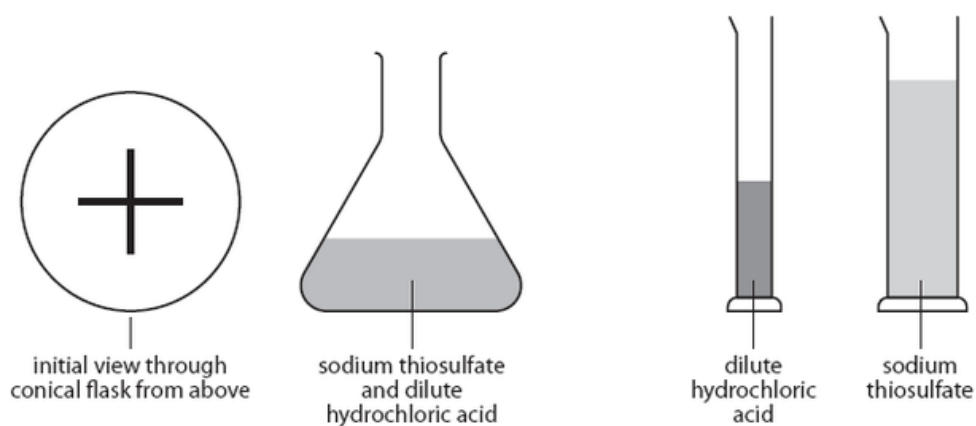
The Kinetic Theory explains the differences between the three states of matter. It states that all matter is made up of moving particles which are molecules or atoms. In solids, the particles are so tightly bound to each other that they can only vibrate but not move to another location.

In liquids, the particles have enough free space to move about, but they still attract one another. In gases, the particles are far apart and can move about freely since there is much free space. Solids change into liquids, and liquids into gases, when the particles gain more kinetic energy, like when being heated and are able to move apart from one another. When the molecules vibrate more quickly upon heating, some of it escapes from the matter. This is what the Kinetic Theory is about.



## PROCEDURE

Put 10 mL of %5 solution of sodium thiosulfate in a flask. Measure 10 mL of %2.5 solution of hydrochloric acid in a small measuring cylinder. Add the acid to the flask and immediately start the clock (at room temperature). Swirl the flask to mix the solutions and place it on a piece of paper marked with a cross. Look down at the cross from above. When the cross disappears stop the clock and note the time. Record this in the table. Repeat these at 35°C, 45°C and 55°C. As soon as possible, pour the solution down the sink (in the fume cupboard if possible) and wash away.



Experiment No	Volume of $\text{Na}_2\text{S}_2\text{O}_3$ (mL)	Volume of HCl (mL)	Temperature	Time taken (s)
1				
2				
3				
4				

## Report Sheet A

Student's Name:

Lab. Instructor name & sign. :

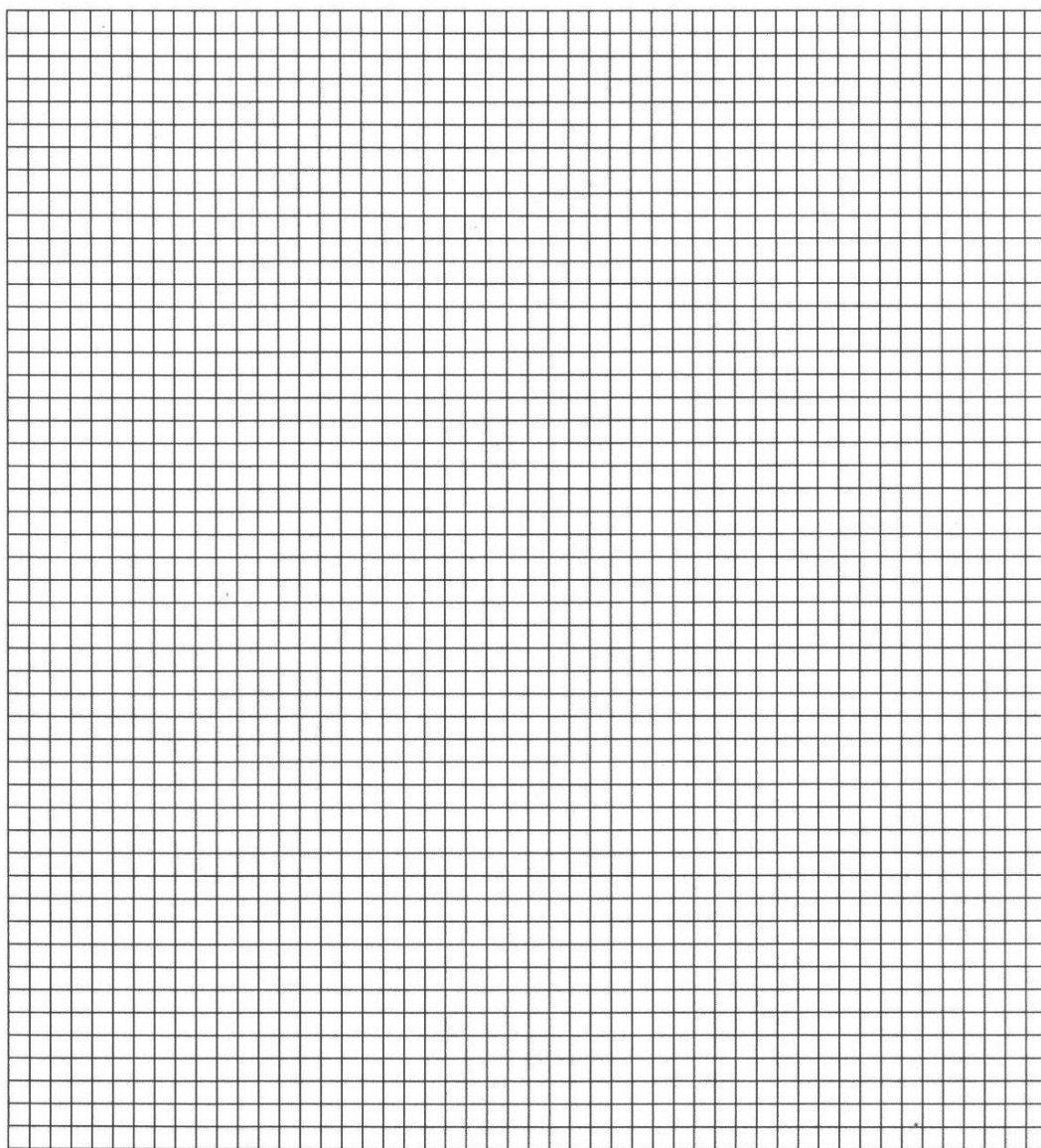
Lab. Section :

Date :

---

Name of the Experiment:

7. Plot a graph of time taken on the vertical (y) axis and  $\text{Na}_2\text{S}_2\text{O}_3$  concentration on the horizontal (x) axis.



**Report Sheet B** (*to be submitted to the instructor*)

**Student's Name:**

**Lab. Instructor name & sign. :**

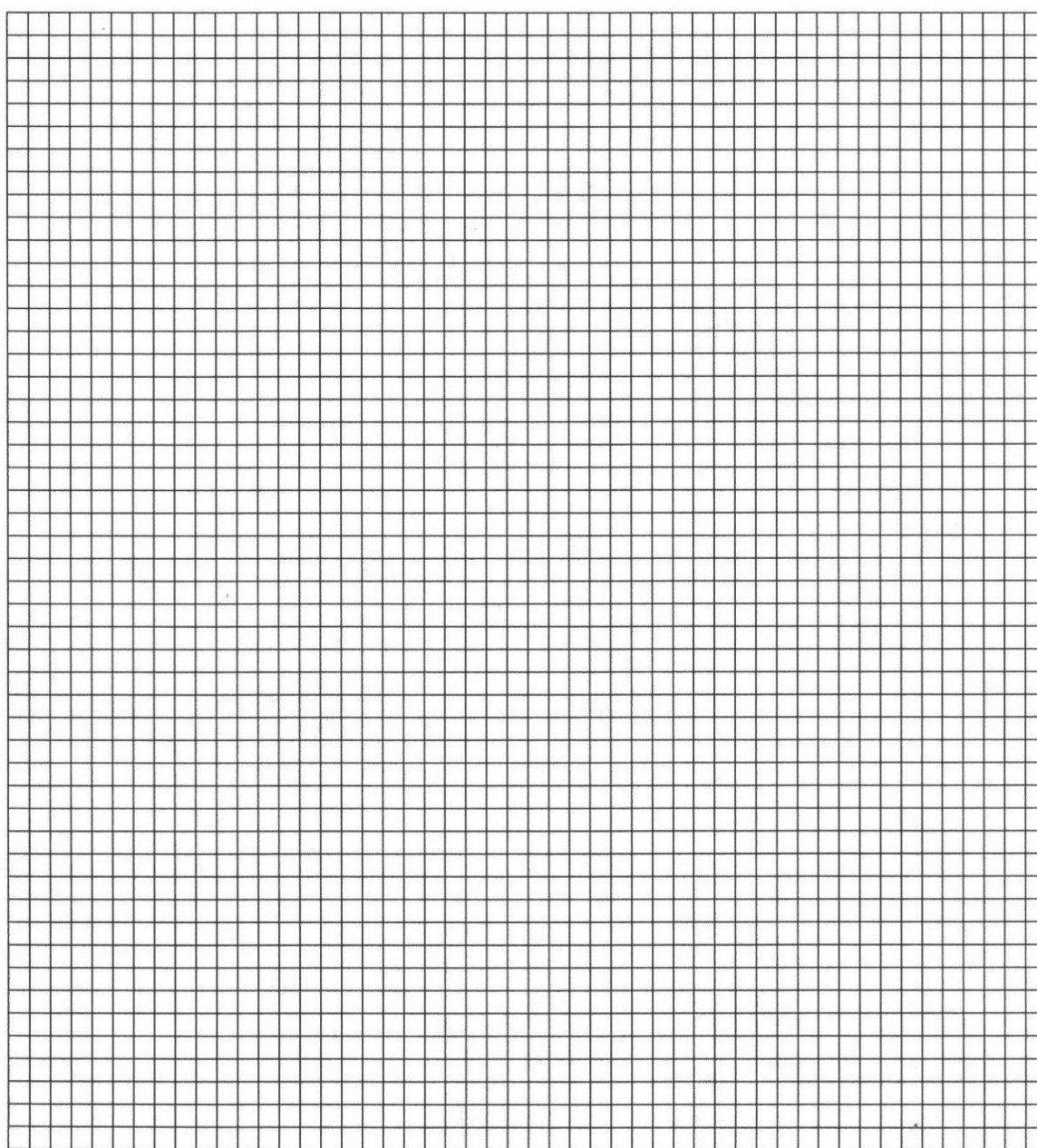
**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

1. Plot a graph of time taken on the vertical (y) axis and  $\text{Na}_2\text{S}_2\text{O}_3$  concentration on the horizontal (x) axis.



## EXPERIMENT NO: 14      EFFECT OF TYPE OF MATTER ON REACTION RATE

### Chemicals and Apparatus

\*Potassium Permanganate,  $\text{KMnO}_4$

\*Hydrogen Peroxide,  $\text{H}_2\text{O}_2$

\*Iron (II) Chloride,  $\text{FeCl}_2$

\*Oxalic Acid,  $\text{H}_2\text{C}_2\text{O}_4$

\*Sulfuric Acid,  $\text{H}_2\text{SO}_4$

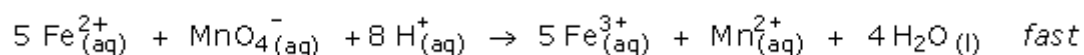
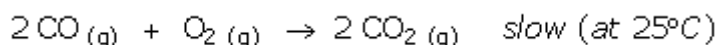
\*Chronometer

### PURPOSE

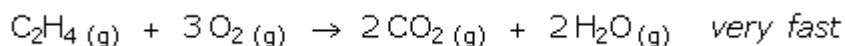
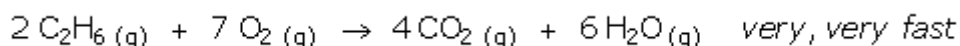
In this experiment the student will study the effect of type of matter on reaction rate.

### THEORY

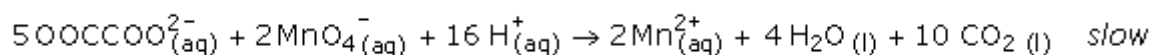
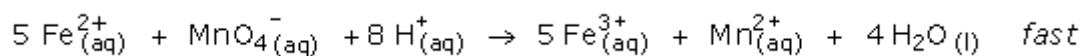
Reactions involving ionic species tend to proceed faster than reactions involving molecular compounds.



Reactions involving the breaking of weaker bonds proceed faster than reactions involving the breaking of stronger bonds. For example, double carbon to carbon bonds are stronger than single C-C bonds.

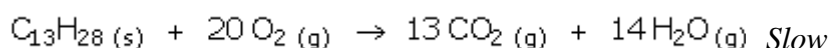
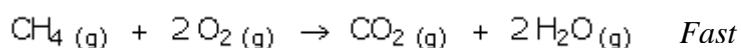


Reactions involving the breaking of fewer bonds per reactant proceed faster than those involving the breaking of a larger number of bonds per reactant.



The simple ion  $\text{Fe}^{2+}$  reacts faster than oxalate ( $\text{OOC}(\text{COO})^{2-}$ ).

Kerosene burns more slowly than methane because there are more bonds to be broken per molecule of kerosene than there are per molecule of methane. Kerosene is a larger molecule



## PROCEDURE

Into three test tubes take 7 mL of 0.1 M  $\text{H}_2\text{O}_2$ , 7 mL of 0.1 M  $\text{FeCl}_2$  and 7 mL of 0.1 M  $\text{H}_2\text{C}_2\text{O}_4$  and to each of these test tubes add 1 mL of conc.  $\text{H}_2\text{SO}_4$  solution. Measure 2 mL of 0.01 M  $\text{KMnO}_4$  solution. Add the  $\text{KMnO}_4$  solution to the first test tube and immediately start the clock. When the pink color disappears stop the clock and note the time. Record this in the table. Repeat these for other three test tubes (note that the test tube including  $\text{H}_2\text{C}_2\text{O}_4$  must be swirled after adding  $\text{KMnO}_4$ ).

Test Tube No	Time taken (s)	Reaction Rate $[\text{KMnO}_4] / \text{time(s)}$
1		
2		
3		

## Report Sheet A

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

8. Calculate the reaction rates and order the reactions by considering the results

9. How do you explain the difference between reaction rates?

**Report Sheet B** (*to be submitted to the instructor*)

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

1. Calculate the reaction rates and order the reactions by considering the results

2. How do you explain the difference between reaction rates?

## EXPERIMENT NO: 15      ACID-BASE TITRATIONS

### Chemicals and Apparatus

\*Sodium Hydroxide, NaOH

\*Hydrochloric Acid, HCl

\*Phenolphthalein Solution

\*Burette

\*Erlenmeyer

\*Flask

### PURPOSE

In this experiment, the student will become familiar with the practical aspects of an acid base titration and indicators. They will learn how to determine the concentration of an unknown acid or base by titration.

### THEORY

Quantitative analysis based on the measurement of volume is called volumetric analysis. These analyses are fairly rapid and give very accurate results. The process of volumetric analysis is called titration. They may be acid-base, precipitation, complexometric and oxidation-reduction titration. **Titration** is the process in which a solution is added drop by drop from the burette into a solution in a flask.

In volumetric analysis, a solution of known concentration is used to determine the unknown concentration of a second solution which reacts with it. A measured volume of the unknown solution is placed in a conical flask, and a solution of known concentration is carefully added dropwise to it until both substances are present in equivalent amounts. At the equivalence point the number of moles of acid is equal to the number of moles of base. An indicator is used to indicate the end point. The end point might differ slightly from the



equivalence point, and the difference between the equivalence point is called the titration error. The titration error will usually be negligible if a suitable indicator is available for use.

The reaction between HCl (a strong acid) and NaOH (strong base) can be represented by



or in terms of net ionic equation by



Thus for a monoprotic acid and base at the end point

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

A standard solution is the one whose concentration is accurately known. In this experiment a sodium hydroxide solution of unknown concentration will be standardized against a standard hydrochloric acid solution. The standardized NaOH solution will be used to determine the molecular weight of a monoprotic acid by titrating a known weight of the acid against the NaOH solution.

**Standardization** is the process to find out the exact molarity of the solution prepared.

**Equivalence Point:** It is the point where the reactants are in stoichiometric ratio. This is a theoretical point, which can not be determined exactly. The aim is to reach the equivalence point as close as possible. To see the equivalence point an indicator is used.

**Indicators** are chemical substances which change color depending on the medium.

**Titration error** is the difference between the end point and the equivalence point.

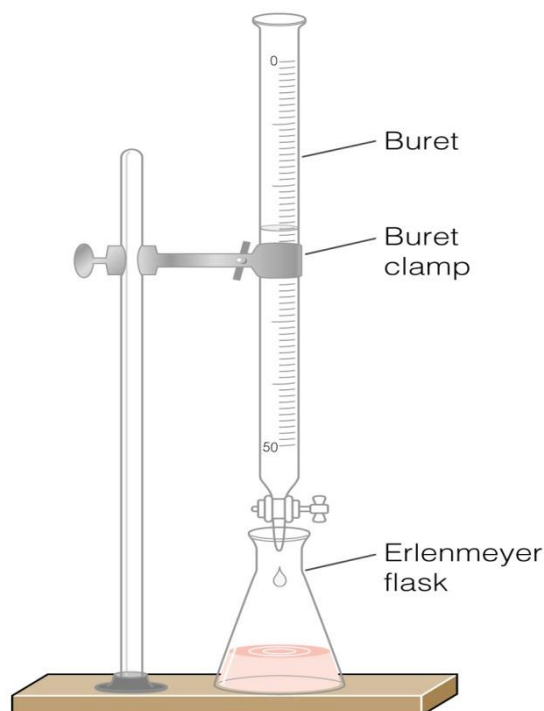
## PROCEDURE

### A. Standardization of NaOH

Take 15 mL of NaOH solution into a clean Erlenmeyer flask and add two drops of phenolphthalein solution. Titrate with HCl until colorless. Record the volume of the HCl solution used. Titrate a second 15 mL sample of the base against the HCl solution, as above. If the two values are more than 0.2 mL apart perform a third titration and discuss the results with your instructor before proceeding further.

### B. Determination of Concentration of an Unknown Acid

Put your unknown sample in a clean Erlenmeyer flask and dilute to 50 mL with water. Add two drops of the phenolphthalein solution. Titrate with the NaOH solution until the pink color of the indicator persists for at least one minute. Record the volume of the NaOH solution used. Titrate the second sample of the unknown acid against the NaOH solution, as above and then the third sample. Calculate the molarity of the acid.



## Report Sheet A

Student's Name:

Lab. Instructor name & sign. :

Lab. Section :

Date :

---

Name of the Experiment:

### A. Standardization of Sodium Hydroxide

1. Molarity of standard HCl solution (Ask your instructor) :
2. Volume of standard NaOH solution :
3. Volume of HCl solution used for the titrations 1<sup>st</sup>:..... 2<sup>nd</sup> :..... 3<sup>rd</sup>:.....

Molarity Of NaOH solution :

### B. Concentration of an Unknown Acid. *Unknown number*:

1. Volume of unknown acid sample :
2. Volume of NaOH solution used for the titrations : 1<sup>st</sup>:..... 2<sup>nd</sup> :..... 3<sup>rd</sup>:.....

Molarity of the unknown acid :

**Report Sheet B** (*to be submitted to the instructor*)

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

**A. Standardization of Sodium Hydroxide**

1. Molarity of standard HCl solution (Ask your instructor) :
2. Volume of standard NaOH solution :
3. Volume of HCl solution used for the titrations 1<sup>st</sup>:..... 2<sup>nd</sup> :..... 3<sup>rd</sup>:.....

Molarity Of NaOH solution :

**B. Concentration of an Unknown Acid. *Unknown number:***

1. Volume of unknown acid sample :
2. Volume of NaOH solution used for the titrations : 1<sup>st</sup>:..... 2<sup>nd</sup> :..... 3<sup>rd</sup>:.....

Molarity of the unknown acid :

## EXPERIMENT NO: 16      pH AND INDICATORS

### Chemicals and Apparatus

- \*Hydrochloric Acid, HCl
- \*Sodium Hydroxide, NaOH
- \*Phenolphthalein
- \*Methyl Orange
- \*Methyl Red
- \*Bromthymol Blue

### PURPOSE

In this experiment you will prepare various solutions of known pH and find out the colors that are characteristics of various indicators in solutions of different pH. You will then use this information to predict the pH values of unknown acid and base.

### THEORY

pH is a logarithmic measure of hydrogen ion concentration, originally defined by Danish biochemist Søren Peter Lauritz Sørensen in 1909.

$$\text{pH} = -\log[\text{H}^+]$$

Where log is a base-10 logarithm and  $[\text{H}^+]$  is the concentration of hydrogen ions in moles per liter of solution. According to the Compact Oxford English Dictionary, the "p" stands for the German word for "power", potenz, so pH is an abbreviation for "power of hydrogen".

The pH scale was defined because the enormous range of hydrogen ion concentrations found in aqueous solutions make using  $\text{H}^+$  molarity awkward. For example, in a typical acid-base titration,  $[\text{H}^+]$  may vary from about 0.01 M to 0.0000000000001 M. It is easier to write "the pH varies from 2 to 13". The hydrogen ion concentration in pure water around room temperature is about  $1.0 \times 10^{-7}$  M. A pH of 7 is considered "neutral", because the concentration of hydrogen ions is exactly equal to the concentration of hydroxide ( $\text{OH}^-$ ) ions

produced by dissociation of the water. Increasing the concentration of hydrogen ions above  $1.0 \times 10^{-7}$  M produces a solution with a pH of less than 7, and the solution is considered "acidic". Decreasing the concentration below  $1.0 \times 10^{-7}$  M produces a solution with a pH above 7, and the solution is considered "alkaline" or "basic".

pH is often used to compare solution acidities. For example, a solution of pH 1 is said to be 10 times as acidic as a solution of pH 2, because the hydrogen ion concentration at pH 1 is ten times the hydrogen ion concentration at pH 2. This is correct as long as the solutions being compared both use the same solvent. You can't use pH to compare the acidities in different solvents because the neutral pH is different for each solvent. For example, the concentration of hydrogen ions in pure ethanol is about  $1.58 \times 10^{-10}$  M, so ethanol is neutral at pH 9.8. A solution with a pH of 8 would be considered acidic in ethanol, but basic in water!

*\*\*\* Hydrochloric acid or muriatic acid is an extremely caustic solution that sits at the extreme end of the acidic scale. This chemical is often used to lower the pH of highly alkaline water, such as in the treatment of swimming pools and aquariums. Only small amounts of hydrochloric acid are required, relative to the amount of water being treated.*

*Also low on the acidic scale at 1.5 – 2.0 is gastric acid, or stomach acids that help us digest food. Colas come in at 2.5, even more acidic than vinegar at about 3.0! It might also be surprising to learn that beer is slightly more acidic than acid rain, and coffee only slightly less acidic.*

*Pure water has a neutral pH and human saliva hovers close to neutral, while our blood is slightly alkaline. Seawater hits the pH scale between 7.7 and 8.3, and products like hand soap, ammonia and bleach score high on the alkaline scale running from 9.0 – 12.5. Highly alkaline baking soda or sodium bicarbonate is often used to increase the base of acidic water.*

## PROCEDURE

Heat to boiling 400 mL of distilled water in a clean flask. Cool with a small beaker inverted over the mouth (Distilled water is usually slightly acidic because of dissolved  $\text{CO}_2$ , which can be expelled by heating). Prepare  $10^{-3}$  M HCl and NaOH solutions for using as stock solutions.

Dilute 5 mL of  $10^{-3}$  M HCl stock solution with 45 mL of boiled distilled water and stir it. The  $\text{H}_3\text{O}^+$  concentration of the final solution is  $10^{-4}$  M, pH= 4.

From 5 mL of pH 4 solution, prepare 50 mL of pH 5 solution and from 5 mL of pH 5 solution, prepare 50 mL of pH 6 solution.

Similarly, dilute the  $10^{-3}$  M NaOH stock solution to prepare successively solutions of pH 10, 9 and 8.

You have now available nine solutions covering the pH range 3 to 11. Place 5 mL portions of these solutions in nine clean dry test tubes and add no more than two drops of methyl orange indicator solution in each test tube. Shake well and observe the color produced in each case. Record the results in the data table on report sheet.

Similarly test the other samples with each of the following indicators (no more than two drops): Phenolphthalein, Methyl Red, Bromthymol Blue.

Place 5 mL of the unknown acid solution (provided from the instructor) in each of five clean, dry test tubes. Add two drops of a different indicator to each test tube. Note the colors obtained.

Proceeding in similar fashion, note the color produced by adding each indicator to the unknown base.

## Report Sheet A

Student's Name:

Lab. Instructor name & sign. :

Lab. Section :

Date

:

---

Name of the Experiment:

**1. Write down the colors obtained for each indicator and pH value**

<i>pH</i>	<i>methyl orange</i>	<i>Phenolphthalein</i>	<i>Methyl Red</i>	<i>Bromthymol Blue</i>
-----------	----------------------	------------------------	-------------------	------------------------

3

4

5

6

7

8

9

10

11

**2. Write down the colors of unknown acid and base obtained for each indicator and pH value**

*Unknown Acid*

*Unknown Base*

*Methyl Orange*

*Phenolphthalein*

*Methyl Red*

*Bromthymol Blue*

**3. Predict the pH values of unknown acid and base.**

pH (unknown acid) :

pH (unknown base) :



**Report Sheet B** (*to be submitted to the instructor*)

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date**

**:**

---

**Name of the Experiment:**

**1. Write down the colors obtained for each indicator and pH value**

<i>pH</i>	<i>methyl orange</i>	<i>Phenolphthalein</i>	<i>Methyl Red</i>	<i>Bromthymol Blue</i>
3				
4				
5				
6				
7				
8				
9				
10				
11				

**2. Write down the colors of unknown acid and base obtained for each indicator and pH value**

	<i>Unknown Acid</i>	<i>Unknown Base</i>
<i>Methyl Orange</i>		
<i>Phenolphthalein</i>		
<i>Methyl Red</i>		
<i>Bromthymol Blue</i>		

**3. Predict the pH values of unknown acid and base.**

pH (unknown acid) :

pH (unknown base) :

## EXPERIMENT NO: 17      TITRATION OF VINEGAR

### Chemicals and Apparatus

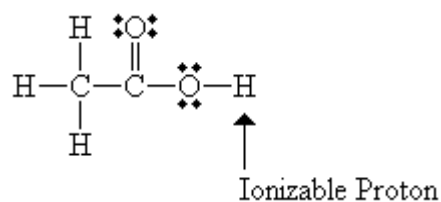
- \*Commercial Vinegar
- \* Phenolphthalein
- \*Sodium Hydroxide, NaOH
- \*Volumetric Flasks
- \*Burette

### PURPOSE

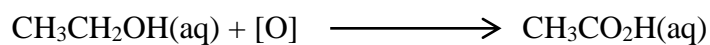
In this laboratory exercise we will determine the percentage Acetic Acid ( $\text{CH}_3\text{COOH}$ ) in Vinegar. We will do this by Titrating the Acetic Acid present with a Strong Base; Sodium Hydroxide (NaOH).

### THEORY

Acetic Acid (fr. Latin *acetum* for vinegar) is the main component of Vinegar. It is a carbon based compound with a single ionizable proton, making it an organic acid of the larger class of organic acids called Carboxylic Acids; organic compounds with a  $-\text{COOH}$  functional moiety.



Alcoholic solutions containing less than 18% Grain Alcohol become Vinegar when airborne bacteria oxidize the Alcohol into Acetic Acid:



(Here, [O] is a general notation for any oxidizing agent.) For instance, Cider Vinegar is produced from fermented apples. Balsamic Vinegar is prepared from the must of white grapes. White Vinegar is prepared from distilled alcohol. Vinegars are used for a variety of purposes; cooking, cleaning, pickling and gardening. Typically the Acetic Acid content of commercial vinegar is 5% (Note that pure Acetic Acid is referred to as Glacial Acetic Acid.)

## PROCEDURE

The vinegar is too concentrated as it is to perform a titration, so make a dilution by putting 5 mL of vinegar (using a pipette) in to a 50 mL volumetric flask and diluting as usual. This dilution means the solution you will be titrating is 10 times weaker than the original vinegar. To take this dilution factor into account, multiply the concentration of diluted sample by 10 to get the concentration of the original vinegar.

Measure out 10 mL of the diluted solution and add a couple of drops of phenolphthalein. Titrate the sample either to the endpoint (the endpoint of the titration will be detected using phenolphthalein indicator; changes color from clear to pink). Perform at least three titrations; If any of the runs are off by more than 0.05 mL from any other run, perform a fourth titration and discard the titration that seems the most significantly disagree with the other runs. Determine the concentration of acid from this step.

**Example:** A student titrates a 25.00 mL sample of vinegar with 1.000 molar NaOH. The volume of base needed to reach the equivalence point is 17.00 mL. What is the concentration of acetic acid in the vinegar in units of grams per 100 mL?

### Solution:

From equation :  $\text{CH}_3\text{COOH} + \text{NaOH} \longrightarrow \text{H}_2\text{O} + \text{CH}_3\text{COONa}$

$$\begin{aligned}\text{moles of acid initially present} &= 1.000 \text{ mol/L} \times 17.00 \text{ mL} \times 1\text{L}/(1000\text{mL}) \\ &= 0.0170 \text{ mol}\end{aligned}$$

This is the number of moles of acetic acid in 25.00 mL of vinegar.

The molecular formula of acetic acid is  $\text{CH}_3\text{COOH}$ . The molar mass is given by; molar mass =  $2 \times 12.0 + 4 \times 1.01 + 2 \times 16.0 = 60.0 \text{ g/mol}$

The gram of acetic acid in 25.00 mL of vinegar is:

$$60.0 \text{ g/mol} \times 0.0170 \text{ mol} = 1.02 \text{ g}$$

1 mL of vinegar contains 1.02 /25 grams per mL

The gram of acetic acid in 100 mL of vinegar is:

$$(1.02 / 25) \text{ g/mL} \times 100 \text{ mL} = 4.08 \text{ g}$$

Therefore, this sample of vinegar meets the federal requirement of a minimum of 4 g of acetic acid per 100 mL of vinegar.

## Report Sheet A

Student's Name:

Lab. Instructor name & sign. :

Lab. Section :

Date :

---

Name of the Experiment:

1. Volume of NaOH solution used for the titrations:

1<sup>st</sup>: .....2<sup>nd</sup>: .....3<sup>rd</sup>: .....4<sup>th</sup> (If necessary): .....

2. Calculate the concentration of acetic acid in the vinegar in units of grams per 100 mL

**Report Sheet B** (*to be submitted to the instructor*)

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

**1.** Volume of NaOH solution used for the titrations:

1st: .....2nd: .....3rd: .....4th (If necessary): .....

**2.** Calculate the concentration of acetic acid in the vinegar in units of grams per 100 mL

## EXPERIMENT NO: 18      CHEMICAL EQUILIBRIUM

### Chemicals and Apparatus

\*Sodium Thiocyanate, SCN

\*Ferric Nitrate, Fe(NO<sub>3</sub>)<sub>3</sub>

\*Ruler

\*Distilled Water

### PURPOSE

In this experiment, the value of the equilibrium constant for the reaction between ferric, Fe<sup>3+</sup> and thiocyanate, SCN<sup>-</sup>, ions will be determined.

### THEORY

Chemical equilibrium has been reached in a reaction when the rate of the forward reaction is equal to the rate of the reverse reaction. When a chemical reaction has reached equilibrium, collisions are still occurring: the reaction is now happening in each direction at the same rate. This means that reactants are being formed at the same rate as products are being formed, and this is indicated by double arrows,  $\rightleftharpoons$ . At equilibrium, the reaction can lie far to the right, meaning that there are more products in existence at equilibrium, or far to the left, meaning that at equilibrium there are more reactants. The concentration of the reactants and products in a reaction at equilibrium can be expressed by equilibrium constant, symbolized  $K$  or  $K_{eq}$ :

For the general reaction

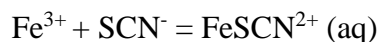


$$\text{Equilibrium constant} = K = \frac{[C]^c[D]^d}{[A]^a[B]^b}$$

The general rule used to predict the direction in which equilibrium will shift is known as the **Le Chatelier Principle**. It states that if a chemical system at equilibrium experiences a

change in concentration, temperature, volume, or partial pressure, then the equilibrium shifts to counteract the imposed change and a new equilibrium is established.

For the reaction between ferric,  $\text{Fe}^{3+}$  and thiocyanate,  $\text{SCN}^-$  ions,



$$\text{The equilibrium constant is, } K = \frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}] [\text{SCN}^-]}$$

To find the value of K, it is necessary to determine the concentration of each of the species  $\text{Fe}^{3+}$ ,  $\text{SCN}^-$  and  $\text{FeSCN}^{2+}$  in the system at equilibrium. This will be done colorimetrically, taking advantage of the fact that  $\text{FeSCN}^{2+}$  is only highly colored species in the solution.

The color intensity of a solution depends on the concentration of the colored species and on the depth of solution viewed. Thus 2 cm of a solution of a 0.1 M colored species appears to have the same color intensity as a 1 cm of a 0.2 M solution. Consequently, if the depths of two solutions of unequal concentrations are chosen so that the solutions appear to be equally colored, then the ratio of concentrations is simply the inverse of the ratio of the two depths. It should be noted that this procedure permits only a comparison between concentrations. To know the absolute values, it is necessary to compare unknown solutions with a standard of known concentration.

In order to determine the concentration of  $\text{FeSCN}^{2+}$ , a standard solution  $\text{FeSCN}^{2+}$  with a known concentration is needed. Such a solution can be prepared by adding a very large excess of  $\text{Fe}^{3+}$  to a solution containing a small and unknown concentration of  $\text{SCN}^-$  so that essentially all the  $\text{SCN}^-$  is converted to  $\text{FeSCN}^{2+}$ . Under these conditions, it can be assumed that the final concentration of  $\text{FeSCN}^{2+}$  is equal to the initial concentration of  $\text{SCN}^-$ .



$$[\text{SCN}^-] \sim [\text{FeSCN}^{2+}]$$



## PROCEDURE

Clean six 15-cm test tubes with distilled water and let them drain. To each of these test tubes add 5 mL of 0.002 M NaSCN. Add 5 mL of 0.2 M  $\text{Fe}(\text{NO}_3)_3$  to the first test tube (This tube will serve as the standard).

For the other test tubes proceed as follows: Add 10 mL of 0.2 M of  $\text{Fe}(\text{NO}_3)_3$  and 15 mL of distilled water into a graduated cylinder to have a 25 mL of diluted solution. Stir thoroughly to mix. Take 5 mL from this solution and pour it into the second test tube.

Discard 10 mL of the diluted solution in the graduated cylinder and add 15 mL of distilled water on it. Stir thoroughly. Take 5 mL from this solution and pour it into third test tube. Discard 10 mL of the solution in the graduated solution in the graduated cylinder and add 15 mL of distilled water. Stir thoroughly. Take 5 mL from this solution and pour it into the fourth test tube. Continue this procedure until you prepare six test tubes.

Now you will determine the concentration of  $\text{FeSCN}^{2+}$  in each test tube relative to the standard in test tube 1. Compare the color intensity of test tube 1 with that of the other test tubes. To do it, take two tubes to be compared, hold them side by side and wrap a strip of white paper around both of them. Look at the solutions from the top. If color intensities appear identical, measure the heights of the solutions in the two tubes being compared. If not, take test tube 1 and pour off some of the standard into a clean beaker (you may need to pour some back) until the color intensities appear identical. Do this comparison for all of the five test tubes.

## CALCULATIONS

For calculation of initial concentrations of  $\text{Fe}^{3+}$  and  $\text{SCN}^-$  :

- assume that both  $\text{Fe}(\text{NO}_3)_3$  and  $\text{NaSCN}$  are completely dissociated in water.
- remember that mixing of two solutions dilute both of them.

For calculation of equilibrium concentrations of  $\text{SCN}^-$ ,  $\text{Fe}^{3+}$ ,  $\text{FeSCN}^{2+}$  :

- for test tube 1, assume that all the initial  $\text{SCN}^-$  has been converted to  $\text{FeSCN}^{2+}$
- for the other test tubes, calculate the equilibrium concentration of  $\text{FeSCN}^{2+}$  from the ratio of heights in the color comparison. Equilibrium concentrations of  $\text{Fe}^{3+}$  and  $\text{SCN}^-$  are obtained by subtracting  $\text{FeSCN}^{2+}$  formed from the initial  $\text{Fe}^{3+}$  and  $\text{SCN}^-$  concentrations.

Calculate the value of K for test tubes 2 to 6. Show your calculation on back of the report sheet for one of the test tubes. Decide which of the value is most reliable.

## Report Sheet A

Student's Name:

Lab. Instructor name & sign. :

Lab. Section :

Date :

Name of the Experiment:

DATA:

Test Tube	2	3	4	5	6
Height of Sample, cm					
Height of standard (test tube 1), cm					

RESULTS

Test Tube	Initial Concentrations		Equilibrium Concentrations			K
	[Fe <sup>3+</sup> ]	[SCN <sup>-</sup> ]	[FeSCN <sup>2+</sup> ]	[Fe <sup>3+</sup> ]	[SCN <sup>-</sup> ]	
1			-	-	-	-
2						
3						
4						
5						
6						

**Report Sheet B** (*to be submitted to the instructor*)

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

**DATA:**

Test Tube	2	3	4	5	6
Height of Sample, cm					
Height of standard (test tube 1), cm					

**RESULTS**

Test Tube	Initial Concentrations		Equilibrium Concentrations			K
	[Fe <sup>3+</sup> ]	[SCN <sup>-</sup> ]	[FeSCN <sup>2+</sup> ]	[Fe <sup>3+</sup> ]	[SCN <sup>-</sup> ]	
1			-	-	-	-
2						
3						
4						
5						
6						

## EXPERIMENT NO: 19      DETERMINATION OF WATER HARDNESS

### Chemicals and Apparatus

\* Ethylenediamine-tetraacetic acid, EDTA

\*Erio Chrome Black T, Erio-T

\*Burette

\*Tap Water

### PURPOSE

This experiment is designed to demonstrate a method to determine the hardness of water samples. It also serves as an example of a method using complex ion formation to do quantitative analysis.

### THEORY

The total concentration of alkaline earth metal ions, such as calcium and magnesium, in water determine the hardness of water. The term hard water comes from the fact that these metal ions precipitate soap molecules from water making it "hard" to get things clean. The calcium in hard water precipitates as calcium carbonate (lime scale), if the water is boiled. Water hardness is usually determined by measuring the total amount of calcium and magnesium present, since the concentrations of these ions far exceed those of other alkaline earth metals. The accepted practices for reporting hardness is as mg  $\text{CaCO}_3/\text{L}$ , as if all of the hardness were from calcium carbonate.

One of the most common applications of complexometric titration is the determination of water hardness. In this application the ions  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  are titrated in  $\text{pH}=10$  with EDTA solution in the presence of erio-T as indicator. Determination of water hardness arising from calcium and magnesium in various water samples can be done easily using EDTA (titriplex-III). Such hardness determination with usage of EDTA is both easy and certain. Several methods have been advanced on this subject. It is also possible to find other techniques in literature. Bonding of  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  in water with the ion  $\text{HCO}_3^-$  causes *temporarily hardness*, with the ions such as  $\text{SO}_4^{2-}$  and  $\text{Cl}^-$  causes *permanent hardness*. Ignoring the species of anion,

the total concentration of  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  cations, which are sources of hardness, in water is known as total hardness (mg  $\text{CaCO}_3$  equivalent/100 mL water) it is also likely to be expressed with other units like French hardness unit ( $^\circ\text{Fr}$ ).

Hardness, mg $\text{CaCO}_3/\text{L}$	Hardness
< 15	very soft
15-50	soft
50-100	medium hard
100-200	hard
> 200	very hard

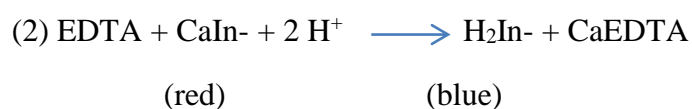
Water Hardness Classification

We will titrate using ethylenediamine-N,N,N',N' tetraacetic acid (EDTA). EDTA is a chelating agent that can donate electrons (Lewis base) thereby forming a complex with metal ions (Lewis acid). The EDTA will complex first with the  $\text{Ca}^{2+}$  and then with the  $\text{Mg}^{2+}$ . As with any titration we will need an indicator to determine when all of the  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  have complexed with the EDTA (i.e. the endpoint). The indicator used in this experiment is Eriochrome Black T. At pH 10 the indicator will be in the form  $\text{HInd}^{2-}$  (Ind stands for indicator), which is blue. The indicator, reacts with  $\text{Mg}^{2+}$  (colorless) to give a red complex.

The general procedure for this experiment starts with a sample of hard water that contains calcium and magnesium. To insure that all cations stay in solution and that the indicator works properly, a buffer is used to adjust the pH to 9.9 - 10.1. After the pH is adjusted and the indicator is added, the EDTA titrant is added via a burette. First the EDTA ( $\text{H}_2\text{Y}^{2-}$ ) will complex with the calcium ions, forming a red solution:



At the endpoint, the EDTA will complex with the calcium and the indicator becomes unbound, which is indicated by the red blue color change:



## PROCEDURE

### A. Solution preparation

**(1) Buffer solution (pH=10):** Dissolve 17.5 g ammonium chloride ( $\text{NH}_4\text{Cl}$ ) in 142 mL concentrated ammonium hydroxide and dilute to 250 mL with distilled water.

**(2) Standard calcium solution:** Place ~ 1.5 g anhydrous calcium carbonate (in oven) into a beaker, and place in a dessicator for 10 minutes. Weigh exactly 1.000g anhydrous calcium carbonate into a clean 600 mL Erlenmeyer flask and add 200 mL deionized water. Add a few drops of 6 M HCl until all  $\text{CaCO}_3$  has dissolved. Add 200 mL distilled water and boil for a few minutes to expel  $\text{CO}_2$ . Transfer quantitatively to a 1000 mL volumetric flask and dilute to the mark with distilled water.

**(3) 0.01 M EDTA Solution :** Dissolve 3.723 g disodium EDTA in distilled water and dilute to 1 L.

### B. Standardization of the EDTA Solution

Before using the EDTA to titrate water samples we must know its exact concentration. We will use the solution of calcium carbonate (1.00 g  $\text{CaCO}_3$ / L) as the primary standard. Measure exactly 15.0 mL of the  $\text{CaCO}_3$  solution into a 250 mL flask. Add approximately 30 mL of deionized water to the flask. Add 2.0 mL of the buffer solution. The remainder of the titration must be completed within 15 minutes of the time when the buffer is added. Add 4 drops of Eriochrome Black T indicator solution. Titrate using the EDTA titrant. At the end point the color should change from red to a pale blue. Repeat this procedure at least twice.

### C. Determination of Permanent Hardness

Measure exactly 25.0 mL of the hard water sample into a 250 mL flask. Add 2.0 mL of the buffer solution. The remainder of the titration must be completed within 15 minutes of the time when the buffer is added. Add 4 drops of Eriochrome Black T indicator solution. Titrate using the EDTA titrant. At the end point the color should change from red to blue. Repeat this procedure at least three times. Use this data and the data from parts A and B to calculate the hardness of your water sample in mg  $\text{CaCO}_3$ /L, ppm (parts per million) and French hardness unit ( $^\circ\text{Fr}$ ).

## CALCULATIONS

$$[\text{Ca}^{2+}] \text{ (Mole/L)} = \frac{(\text{M}_{\text{EDTA}}) \times (\text{V}_{\text{EDTA}})}{\text{V}_{\text{water sample (mL)}}$$

$$\text{ppm CaCO}_3 = \text{mg CaCO}_3/\text{L}$$

$$\text{ppm CaCO}_3 = [\text{Ca}^{2+}] \times \frac{100 \text{ g CaCO}_3}{1 \text{ mole of CaCO}_3} \times \frac{1000 \text{ mg}}{1 \text{ g}}$$

$$\text{French hardness (°Fr)} = 10 \text{ ppm CaCO}_3/\text{L}$$

### ***The Negative Effects of Hard Water***

*Hard water interferes with almost every cleaning task from laundering and dish washing to bathing and personal grooming. Clothes laundered in hard water may look dingy and feel harsh and scratchy. Dishes and glasses may be spotted when dry. Hard water may cause a film on glass shower doors, shower walls, bathtubs, sinks, faucets, etc. Hair washed in hard water may feel sticky and look dull. Water flow may be reduced by deposits in pipes.*

*Dealing with hard water problems in the home can be a nuisance. The amount of hardness minerals in water affects the amount of soap and detergent necessary for cleaning. Soap used in hard water combines with the minerals to form a sticky soap curd. Some synthetic detergents are less effective in hard water because the active ingredient is partially inactivated by hardness, even though it stays dissolved. Bathing with soap in hard water leaves a film of sticky soap curd on the skin. The film may prevent removal of soil and bacteria. Soap curd interferes with the return of skin to its normal, slightly acid condition, and may lead to irritation. Soap curd on hair may make it dull, lifeless and difficult to manage.*



## Report Sheet A

Student's Name:

Lab. Instructor name & sign. :

Lab. Section :

Date :

---

Name of the Experiment:

1. Volume of EDTA solution used for titrations 1<sup>st</sup>: .....2<sup>nd</sup>: .....3<sup>rd</sup>: .....
2. Calculate the permanent hardness value in terms of ppm  $\text{CaCO}_3$  and French Hardness ( $^{\circ}\text{Fr}$ ).

$[\text{Ca}^{2+}]$  (Mole/L) =

ppm  $\text{CaCO}_3$  =

French hardness ( $^{\circ}\text{Fr}$ ) =

**Report Sheet B** (*to be submitted to the instructor*)

**Student's Name:**

**Lab. Instructor name & sign. :**

**Lab. Section :**

**Date :**

---

**Name of the Experiment:**

1. Volume of EDTA solution used for titrations 1<sup>st</sup>: .....2<sup>nd</sup>: .....3<sup>rd</sup>: .....
2. Calculate the permanent hardness value in terms of ppm CaCO<sub>3</sub> and French Hardness (°Fr).

[Ca<sup>2+</sup>] (Mole/L) =

ppm CaCO<sub>3</sub> =

French hardness (°Fr) =

## REFERENCES

1. Experiments in General Chemistry, Chemistry Department of Middle East Technical University, 2005
2. Laboratory Experiments in General Chemistry, Eliza Kalvo, Bogazici University, 2008
3. General Chemistry Laboratory Manual, Richard E. Bleil, Dakota State University, 2005
4. General Chemistry Laboratory, Nermin Uysal, Marmara University, Atatürk College of Education, 1995
5. Experiments in General Chemistry, Gerald S. Wess at all., Sixth Edition, 1993
6. Experiments for General Chemistry, A.A. Russell, E.D. Siebert, Burges International Group, 1991
7. Experiments in General Chemistry, Thomas Greco, Lyman H. Rickard, Gerald Weis, Prentice Hall, 2002