

GENERAL CHEMISTRY LABORATORY MANUAL (2024-2025)

LABORATORY RULES FOR EKİM110 GENERAL CHEMISTRY LAB.

- 1. The students must attend the General Chemistry Laboratory course in exact day, time and location, which are announced on the curriculum of the departments.
- 2. Attendance compulsion is 70% for the laboratory semester. If a student does not attend for at least 2 experiments, a VF will be issued.
- 3. During the lab course, students have to wear their lab coats, lab goggles and latex gloves. The students must bring their own lab coat, lab goggles and latex gloves to the lab. Be aware that failing to wear lab coat, lab goggles and latex gloves in the laboratory will result in immediate expulsion from the laboratory, failing the experiment. This rule is strictly enforced.
- **4.** The student must be on the lab 10 min before the experiment starts. Afterwards, she/he will not to be allowed to attend the lab and perform the experiment.
- **5.** During the lab course, it is strictly forbidden to use mobile phones. The students must not leave the lab and experiment set without permission of the lab assistant.
- **6.** In order to provide life safety and to ensure the success of experiment in a safe manner, hand jokes among the students are strictly forbidden. Also, touching to chemicals without gloves, sniffing and tasting them are dangerous.
- 7. In each experiment, required chemicals and materials will be supplied to the students by lab assistants. The students will not borrow the chemicals and materials from the other students. At the end of each experiment, all the glass or metal materials will be cleaned by the students. Also, the experiment set will be tidy and clean.
- **8.** Before coming to the lab, the students are expected to read the experiment of the week in this booklet thoroughly and to know about the procedure of the regarding experiment and theoretical information.
- **9.** At the start of each laboratory, the students will be given a prelab quiz. The object of this quiz is to assess whether they are well prepared for the experiment.
- **10.** The total grade of an experiment will be the sum of the report grade (%60) and quiz grade (%40).
- **11.** At the beginning of the semester, the students will be informed about the safety rules of laboratory.

Reference: H. Güler, D. Saraydın, U. Ulusoy, Genel Kimya Laboratuvarı, Hatipoğlu yayınları, Ankara, 1993

<u>These rules were taken from the booklet of Eskişehir Technical University, Faculty of Science, Department of Chemistry,</u>
General Chemistry Laboratory.

LABORATORY SAFETY RULES AND CAUTIONS

- 1. Lab coats must be worn at all times when in the laboratory! All students have to wear lab coat and students without lab coat will not be allowed in the lab.
- 2. Safety glasses must be worn at all times when in the laboratory! All students have to wear safety glasses in the lab and students without safety glasses will not be allowed in the lab. Contact lenses are not allowed in the lab because acid and organic chemical vapors could get in between eye and lenses, lenses can be glued on the eye in the case of an accident and can be difficult to remove.
- **3. Dress appropriately!** Wide dresses, sandal type shoes shouldn't be worn in the lab and long hair should be tied.
- 4. No Foods or Drinks! Food, drink and chewing gum are not allowed in the lab.
- **5. Know the location of exits and safety equipment!** All students should know where the fire extinguisher, first aid cabinet and shower are placed in the lab.
- **6.** Learn the speediest exit from the lab. in the case of a fire.
- 7. Use the shower when your dresses or hair catches fire.
- **8.** Do not run and do not make jokes in the chemistry lab.
- 9. Benches should not be used to sit on or to leave bags or personal things.
- 10. Smoke and vapor released during chemical reactions should not be exposed directly.
- 11. Do not attempt unauthorized experiments! It is not allowed to work in chemistry labs. without attendance of assistants or instructors.
- **12.** Do not use Bunsen burners next to flammable chemicals (e.g. ethers)
- **13.** Read the labels carefully on the bottle before use of any chemicals.
- 14. Read the experimental procedure before coming to the lab. Students who come to lab having no knowledge about experiment could create risks for themselves and other students.
- **15.** In the case of any accident (glass cut, acid/base burn, fainting etc.), know where to get help fast and immediately inform your assistant or instructor.
- **16.** Do not orientate the test tube toward yourself and your friend. Reaction carried out in the test tube could be dangerous.
- **17.** Water should not be added on concentrated acids. Acids should be added to the water slowly and by stirring.

- **18.** It is forbidden to smell, to taste the chemicals or to pull solutions by mouth when using pipet. Immediately clean up any chemicals that you spill. If necessary, obtain advice on the cleanup procedure from the lab assistant.
- **19.** Chemicals (solid, liquid or solution) must not be dumped into the sink. Waste bottles in the lab should be used. (Learn where the waste bottles in the lab are.)
- 20. Use the "broken glass labeled container in the lab for the broken glass pieces.
- 21. Matchstick, litmus paper must not be disposed to the sink.
- **22.** Mercury vapor is invisible and toxic. Mercury in the broken thermometer is very dangerous. You must inform your assistant in the case of thermometer breakage.
- **23.** Materials like hot test tube, crucible, and beaker must not hold by hand. They should be cautiously held by tube tongs or left to cool on an asbestos wire.
- **24.** Please use the amounts of chemicals given in the procedure. Use of excess amounts can make difficult to control the reactions or cause the side reactions.
- **25.** The chemicals left behind the experiment should not be returned to the stock bottles, should be discarded into the waste bottle.
- **26.** Always keep your working area, the balance and its environment clean. Work clean and tidy in the lab.
- **27.** Don't change the locations of the stock chemicals during the experiment.28. At the end of the experiment, hand in all the materials you used to your assistant as cleaned.
- **28.** Before leaving the lab, make sure that gas and water taps are closed (turned off).
- **29.** Wash your hands before leaving the lab.

Reference: H. Güler, D. Saraydın, U. Ulusoy, Genel Kimya Laboratuvarı, Hatipoğlu yayınları, Ankara, 1993

<u>These rules were taken from the booklet of Eskişehir Technical University, Faculty of Science, Department of Chemistry,</u>
General Chemistry Laboratory.

SOME LABORATORY EQUIPMENTS



beaker



erlenmayer flask



round-bottom flask



volumetric flask



Tripod



extention clamp



T-connector tube



funnel



filter flask



Wire gauze



Receiver Adapter



Receiver Adapter with vacuum



seperating funnel



test tubes



Watch glass



Mortar and pestle (porcelain)



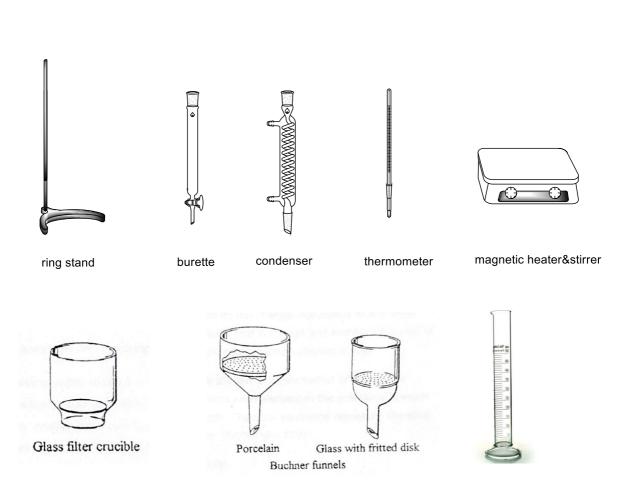


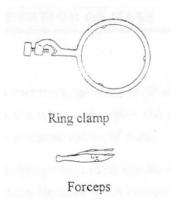


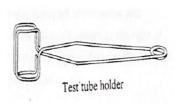
Test tube brush

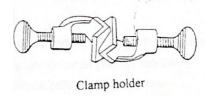


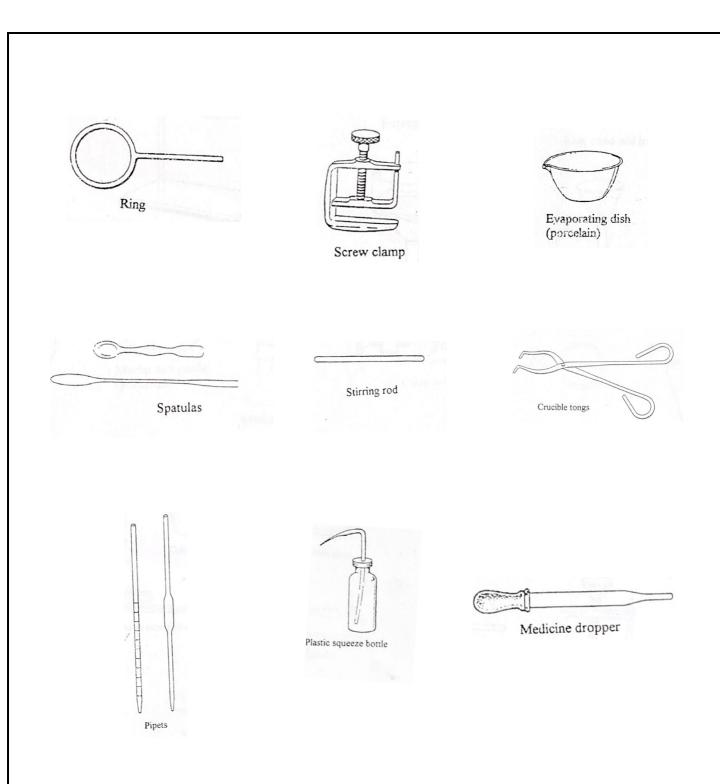
Crucible and cover











Reference: H. Güler, D. Saraydın, U. Ulusoy, Genel Kimya Laboratuvarı, Hatipoğlu yayınları, Ankara, 1993

<u>These laboratory equipments were taken from the booklet of Eskişehir Technical University, Faculty of Science,</u>

<u>Department of Chemistry, General Chemistry Laboratory.</u>

WORK SCHEDULE

Week No.	Experiment Title	Group A-B Thursday 11.00-13.00	Group C-D Friday 09.00-11.00
1	Laboratory Rules and Safety https://youtu.be/4 AUi5nFwmQ https://youtu.be/s9etU3XfxB0 Introduction of Basic Laboratory Materials and Applications https://youtu.be/xy1sv6KGjeE		21.02.2025
2	Identification of Substances According to Their Properties https://bit.ly/2Bu4Xhz	27.02.2025	28.02.2025
3	Solution Preparation https://youtu.be/GVNwg0pkdR8	06.03.2025	07.03.2025
4	Determination of Molecular Mass of Volatile Liquids	13.03.2025	14.03.2025
5	Thermal Decomposition of Potassium Chlorate and Determination of the Molar Volume of Oxygen	20.03.2025	21.03.2025
6	Intermolecular Interactions: Dissolution of alcohols in water and ether https://youtu.be/7sHI0mbhW7Q	27.03.2025	28.03.2025
	RAMADAN FEAST-BANK HOLIDAY (March 29-A	pril 1, 2025)	
	FIRST WEEK OF MIDTERMS		
9	Colligative Properties: Freezing point depression and boiling point elevation https://www.youtube.com/watch?v=eqnmSWcOJpQ	17.04.2025	18.04.2025
	APRİL 23, NATIONAL SOVEREIGNTY AND CHIL	DRENS' DAY	
	MAY 1, LABOUR DAY-BANK HOLIDA	Y	
12	Titration of Weak Acids, pH and Buffers https://youtu.be/pZNU35qWTXM	08.05.2025	09.05.2025
13	Investigation of Factors Affecting Reaction Rate https://youtu.be/C4EHrGv7Sn8	15.05.2025	16.05.2025
14	Chemical Equilibrium and Determination of Equilibrium Constant https://youtu.be/UaP-60FYsD8	22.05.2025	23.05.2025
15	Redox Reactions: Investigation of metal activities	29.05.2025	30.05.2025
15	Make-up	29.05.2025	30.05.2025

IDENTIFICATION OF SUBSTANCES ACCORDING TO THEIR PORPERTIES

MATERIALS AND REAGENTS

• Copper sulfate (CuSO₄) Nitric acid (HNO₃)

• Sodium sulfate (Na₂SO₄) Barium nitrate (Ba(NO₃)₂)

• Barium sulfate (BaSO₄) Sulfuric acid (H₂SO₄)

• Sodium carbonate (Na₂CO₃) Test tube

Magnesium oxide (MgO)
 Distilled water

• Starch (C₆H₁₀O₅) Porcelain

• Sugar $(C_6H_{12}O_6)$

CAUTIONS

Do not smell, taste and touch to the chemicals in the lab. Both acids and bases can be corrosive to human tissue. When concentrated, they can react with tissue and break it down. In general, the more concentrated the acid or base happens to be, the more hazardous it is. Although the more concentrated acids and bases are the most dangerous ones, don't ignore the dilute ones.

You must be particularly careful about getting them in your eyes. The best way to avoid this is to wear safety glasses or goggles when handling either acids or bases. If you do get any in your eyes, let the instructor know and flush it out immediately with lots of water, several minutes worth. There are eye washes in the lab. If you have not yet learned where they are, and how to use them, ask for instructions now or the next time you are in the lab.

Suppose you get some acid or base on you, other than in your eyes. The procedure is the essentially the same: flush that area immediately for several minutes with water and consult the instructor for further advice. If you should be unfortunate enough to spill it all over you, use the safety shower in the lab.

If you spill acid or base on the lab bench top or on the floor, treat it immediately. If it is an acid, first neutralize it with sufficient sodium bicarbonate, (commonly known as baking soda). We have some readily available in the lab or in the adjoining prep room. When the baking soda no longer bubbles when you work it into the spill, the acid is neutralized and you can clean up the mess. If it's a base, you can neutralize it with some vinegar, which is dilute acetic acid. In any case, clean up the area thoroughly. We also have spill kits for use

with extensive spills. The quantities and concentrations of acids and bases used in the exercises and demonstrations in this lesson are permissible to flush down the drain. However, the quantity involved in a spill should be neutralized before disposal.

<u>PURPOSE</u>To examine seven substances and to observe enough properties of each so it can be distinguished from the other six. Then, students will be given an unknown and asked to identify it. Also, to learn about solutions and precipitates both by reading the theoretical part and then do the experiments.

INTRODUCTION AND THEORY

Every substance has a large number of physical and chemical properties. Physical properties include color, smell, taste, solubility, density, electrical conductivity, heat conductivity, melting and boiling points. When a physical property is observed, the substance retains its chemical identity.

In contrast to physical properties, when chemical properties are observed, new substances are formed. Chemical properties include decomposition by heating, and reactions of the substance with water, oxygen, acids, bases, etc.

Solutions: When a solid is mixed with a liquid, the resultant mixture may still be another liquid containing no visible solid particles although its color now may be different.

For the process described above, the resultant mixture called as *solution*, the solid used is a *solute*, and the liquid originally used is *solvent*. When a solution forms, we can state that "The solid dissolves in this liquid" or "The solid is soluble in this liquid". CuSO₄ (solute), is soluble in water (solvent) and the solution formed is blue. After mixing a solid and a liquid, if the solid remains in its original physical form, we state that "The solid does not dissolves in this liquid" or "The solid is INSOLUBLE in this liquid".

EXAMPLE 1:

MgO is insoluble in water.

We have described the solid-liquid solution above which is the most common type. However, it must be known that there are other types of solutions also.

Precipitates: Sometimes, when two solutions are mixed, a new substance may form (a chemical change) which is also insoluble in the solvent used. The solid that forms in this manner is called a *precipitate* (ppt) and the process is manned as *precipitation*.

EXAMPLE 2:

When water solutions of Na₂SO₄ and Ba(NO₃)₂ are mixed, BaSO₄ precipitates.

$$Na_2SO_4(aq) + Ba(NO_3)_2(aq) \rightarrow BaSO_4(s) + 2Na^+ + 2NO_3^-$$

White ppt

PROCEDURE

- During solubility tests, be sure that you have sufficiently shaken the test tube and waited long enough before deciding on solubility characteristics.
- After examining behavior on heating, cool hot porcelain pieces under flowing cold tap
 water before putting them on any surface to prevent FIRE & BURNS. Also use tongs
 while heating the porcelain pieces.
- 1. Prepare seven pieces of paper and write the names of the solid substances mentioned in the special items part.
- 2. Obtain about half a teaspoon of each of the solid substances and place each sample on the proper piece of paper.
- **3.** Take small samples of each substance placed on piece of papers and carry out the following tests on each.

TESTS

1. Solubility in Water

- 1. Fill a test tube to the half with distilled water and add a sample of the substance.
- 2. Shake the test tube several times and observe whether or not the substance dissolves. Some substances dissolve slowly, so you may have to wait a few minutes to be sure.
- 3. Record all properties such as color, gas evolution, etc.

2. Behavior on Heating

- 1. Place a small quantity of a substance on a clean piece of porcelain and heat moderately (see Figure 2.1).
- 2. Record all changes like color, gas evolution, melting, odor, etc.

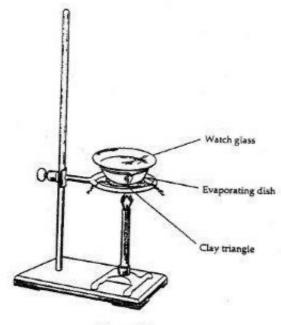


Figure 2.1

3. Solubility in Nitric Acid (HNO₃)

- 1. Fill a test tube to the half with dilute HNO₃ ant test the solubility of the substances as you did with water in test 1.
- **2.** If substance is insoluble in nitric acid, stop at this pint; do not proceed with tests 4 and 5.
- **3.** If the substance is soluble in HNO₃, divide solid containing acid solution equally into two test tubes first to be used in test 4 the second to be used in test 5.

4. Reaction with Barium Nitrate (Ba(NO₃)₂) Solution

- 1. Add equal volume of barium nitrate solution to the first half you obtained in test 3.
- 2. State formations of the precipitate if occur and write the color of it.

5. Reaction with Sulfuric Acid (H₂SO₄)

- 1. Add an equal volume of dilute sulfuric acid to the second half from test 3.
- 2. State formations of the precipitate if occur and write the color of it.

6. Identification of Unknown

- 1. Now, you will be given an unknown that is actually one of the seven substances you used above. Take your unknown from your assistant and identify it.
- 2. When you have identified your unknown, write on your data sheet.
- **3.** For preparation of data sheet, use the notation below:

SOLUBLE : S INSOLUBLE : I

PRECIPITATE FORMATION : PPT

NO PRECIPITATE FORMATION : NO PPT

COLOR CHANGE : CC (Write new color of substance)

GAS FORMATION : GAS

BURNING : **B (Write color of burned substance)**

This experimental procedure was taken from the booklet of Eskişehir Technical University, Faculty of Engineering, Department of Chemical Engineering, General Chemistry Laboratory.

Report Sheet

IDENTIFICATION OF SUBSTANCES ACCORDING TO THEIR PROPERTIES

Student's Name	:	Date:
Group No	:	

	Solubility in water	Behavior on heating	Solubility in HNO ₃ soln.	Reaction with Ba(NO ₃) ₂ soln.	Reaction with H ₂ SO ₄ soln.
CuSO ₄					
Starch					
Na ₂ CO ₃					
BaSO ₄					
Na ₂ SO ₄					
Sugar					
MgO					
Unknown					

Un	known	sample	is	S	

SOLUBLE : S
INSOLUBLE : I

PRECIPITATE FORMATION : PPT

NO PRECIPITATE FORMATION : NO PPT

COLOR CHANGE : CC (Write new color of substance)

GAS FORMATION : GAS

BURNING : B (Write color of burned substance)

QUESTIONS

- 1. How would you distinguish a sample of Na₂CO₃ from a sample of Na₂SO₄?
- 2. If you had three unknowns which are starch, BaSO₄, and MgO, how can you identify starch among them?

SOLUTION PREPARATION

MATERIALS AND REAGENTS

- $K_2Cr_2O_7$
- Concentrated HCl solution
- Analytical balance
- Beakers (100 mL)
- Volumetric flask (50 mL)
- Graduated cylinder (25 mL)
- Pipettes (5 and 25 mL)
- Watch glass
- Stirring rod

PURPOSE

To prepare solutions with known concentrations.

INTRODUCTION AND THEORY

In chemistry, a solution is a homogeneous mixture composed of two or more substances. In such a mixture, a solute is dissolved in another substance, known as a solvent. A common example is a solid, such as salt or sugar, dissolved in water, a liquid. Gases may dissolve in liquids, for example, carbon dioxide or oxygen in water. Liquids may dissolve in other liquids. All solutions are characterized by interactions between the solvent molecules and solute molecules or ions that result in a net decrease in free energy.

Many types of solutions exist, as solids, liquids and gases can be both solvent and solute, in any combination:

Table 1. Various kinds of solutions

Examples of solutions			Solute			
		Gas	Liquid	Solid		
	Gas	Oxygen and other gases in nitrogen (air)	Water vapor in air	Naphthalene slowly sublimes in air, going into solution.		
Solvent	Liquid	Carbon dioxide in water (carbonated water)	Ethanol (common alcohol) in water; various hydrocarbons in each other (petroleum)	Sucrose (table sugar) in water; sodium chloride (table salt) in water; gold in mercury, forming an amalgam		
	Solid	Hydrogen dissolves rather well in metals such as in platinum.	Hexane in paraffin wax, mercury in gold.	Steel, duralumin, other metal alloys		

In chemistry, concentration is the measure of how much of a given substance there is mixed with another substance. This can apply to any sort of chemical mixture, but most frequently the concept is limited to homogeneous solutions, where it refers to the amount of solute in the solvent.

For scientific or technical applications, a qualitative account of concentration is almost never sufficient; therefore quantitative measures are needed to describe concentration. There are a number of different ways to quantitatively express concentration; the most common are listed below.

<u>Molarity</u>: (in units of mol/L, molar, or M) or molar concentration denotes the number of moles of a given substance per liter of solution. A capital letter M is used to abbreviate the units of mol/L. For instance:

(2.0 moles of dissolved particles) / (4.0 liters of solution) = 0.5 mol / L

Such a solution may be described as "0.50 molar." It must be emphasized that a 0.5 molar solution contains 0.5 moles of solute in 1.0 liter of *solution*. This is *not* equivalent to 1.0 liter of *solvent*. A 0.5 mol/L solution will contain either slightly more or slightly less than 1 liter of solvent because the process of dissolution causes the volume of the liquid to increase or decrease.

Molality: (mol/kg, molal, or m) denotes the number of moles of solute per kilogram of solvent (not solution). The 1.0 molal solution is a solution which contains one mole of the solute per 1000 grams of the solvent. For instance: adding 1.0 mole of solute to 2.0 kilograms of solvent constitutes a solution with a molality of 0.50 mol/kg. Such a solution may be described as "0.50 molal".

The mole fraction x, (also called molar fraction) denotes the number of moles of solute as a proportion of the total number of moles in a solution. For instance: 1 mole of solute dissolved in 9 moles of solvent has a mole fraction of 1/10 or 0.1. Mole fractions are dimensionless quantities. (The mole percentage or molar percentage, denoted "mol %" and equal to 100% times the mole fraction, is sometimes quoted instead of the mole fraction.)

Mass percentage (fraction) denotes the mass of a substance in a mixture as a percentage of the mass of the entire mixture. (Mass fraction x_m can be used instead of mass percentage by dividing mass percentage to 100.) For instance: if a bottle contains 40 grams of ethanol and 60 grams of water, then it contains 40% ethanol by mass or 0.4 mass fraction ethanol. In older texts and references this is sometimes referred to as weight-weight percentage (abbreviated as w/w or wt%).

Mass-volume percentage: (sometimes referred to as weight-volume percentage or percent weight per volume and often abbreviated as m/v% or w/v%) describes the mass of the solute in g per 100 mL of the resulting solution. Mass-volume percentage is often used for solutions made from a solid solute dissolved in a liquid. For example, a 40% w/v sugar solution contains 40 g of sugar per 100 mL of resulting solution.

Volume-volume percentage: (sometimes referred to as percent volume per volume and abbreviated as v/v%) describes the volume of the solute in mL per 100 mL of the resulting solution. This is most useful when a liquid-liquid solution is being prepared, although it is

used for mixtures of gases as well. For example, a 40% v/v ethanol solution contains 40 mL ethanol per 100 mL total volume.

<u>Normality</u>: expressed in gram equivalent weights of solute per liter of solution. The definition of a gram equivalent varies depending on the type of chemical reaction that is discussed. As ions in solution can react through different pathways, there are three common definitions for normality as a measure of reactive species in solution:

- In acid-base chemistry, normality is used to express the concentration of protons or hydroxide ions in the solution. Here, the normality differs from the molarity by an integer value each solute can produce n equivalents of reactive species when dissolved. For example: 1 M aqueous Ca(OH)₂ is 2 N (normal) in hydroxide.
- In redox reactions, normality measures the quantity of oxidizing or reducing agent that can accept or furnish one mole of electrons. Here, the normality scales from the molarity, most commonly, by a fractional value. Calculating the normality of redox species in solution can be challenging.
- In precipitation reactions, normality measures the concentration of ions which will precipitate in a given reaction. Here, the normality scales from the molarity again by an integer value.

<u>Parts-per notation:</u> is used in some areas of science and engineering because it does not require conversion from weights or volumes to more chemically relevant units such as normality or molarity. It describes the amount of one substance in another. It is the ratio of the amount of the substance of interest to the amount of that substance plus the amount of the substance it is in.

- *Parts per million* (ppm) denotes the amount of a given substance in a total amount of 1,000,000 regardless of the units of measure used as long as they are the same. e.g. 1 milligram per kilogram. 1 part in 10⁶.
- *Parts per billion* (ppb) denotes the amount of a given substance in a total amount of 1,000,000,000 regardless of the units of measure as long as they are the same. e.g. 1 milligram per tonne. 1 part in 10⁹.

PROCEDURE

PART A: Preparation of 5% (w/w%) K₂Cr₂O₇ solution

K₂Cr₂O₇

Molecular Weight: 294.18 g mole⁻¹ Weight percentage: 99%

- 1. Before starting the experiment, first calculate how many grams of K₂Cr₂O₇ you need to prepare 20 g aqueous solution which contains 5% K₂Cr₂O₇. After that, show your results to responsible person in your laboratory.
- 2. Carefully weigh K₂Cr₂O₇ that you need on a watch glass by using balance and transfer it into 100 mLbeaker.
- 3. Dissolve the K₂Cr₂O₇ with required amounts of water calculated before.
- **4.** The solution stirred with a stirring rod up to all solid particles dissolve.
- 5. Calculate the molality of the prepared solution.

PART B: Preparation of 0.1 M of 50 mL HCl solution

Since HCl is gas at room temperature, we use concentrated HCl acid solution in the laboratory. This concentrated solution is called stock solution. On the label of HCl solution, the following information is given.

HCl

Molecular Weight: 36.5 g/mol Density: 1.2 g/ml Weight percentage: 36.5 %

- 1. Before starting the experiment, first calculate how many moles of HCl you need. At the calculation find the number of moles HCl from givens. (0.1 M of 50 mL HCl)
- 2. After finding number of moles HCl, calculate the mass of HCl that you need.
- 3. Percentage of the stock solution is given (36.5 %). Calculate how many grams of stock solution contains grams (your result at number 2) of HCl.
- 4. Now you found mass of stock solution that you need. Find the volume of this solution that you need. (density:1.2 g/ml)
- 5. By using a pipette, take ___ ml (your result at number 4) stock solution and transfer into 50 mL volumetric flask.
- **6.** Add water till the volume becomes 50 mL.
- 7. Your solution is 50 mL and 0.1 M now.
- **8.** Calculate the density of the prepared solution.

9. Calculate the molality of the prepared solution.

Reference: H. Güler, D. Saraydın, U. Ulusoy, Genel Kimya Laboratuvarı, Hatipoğlu yayınları, Ankara, 1993

This experimental procedure was taken from the booklet of Eskişehir Technical University, Faculty of Science, Department of Chemistry, General Chemistry Laboratory.

QUESTIONS

- 1. What is the main difference between the concentration units "molarity" and "molality"?
- 2. 10 mL of ethanol is dissolved in water to produce a 100 mL of solution. The concentrations expressed in volume percent and mass percent ethanol are 10.00% and 8.03%, respectively, indicating that the mass percent is lower than the volume percent. Explain why this is also true for all aqueous solutions of ethanol. Would it be true of all ethanol solutions, regardless of the other component? Explain.
- **3.** A certain brine has 3.87% NaCl by mass. A 75.0 mL sample weighs 76.9 g. How many liters of this solution should be evaporated to dryness to obtain 725 kg NaCl?

DETERMINATION OF MOLECULAR MASS OF VOLATILE LIQUIDS

MATERIALS AND REAGENTS

- Aluminium foil
- Unknown liquid
- Water

CAUTIONS

- Most unknowns are flammable. Use a moderate flame or heating
- Dispose of the leftover unknown liquid in the "waste liquids" container

PURPOSE

To determine the Formula mass (molecular weight) of a volatile liquid

INTRODUCTION AND THEORY

The measurement of the Formula mass of a compound is one of the first determinations a chemist makes after the synthesis of a new compound. In fact, in working with chemicals in academia, research and industry, chemists must always know the properties of the chemicals with which they are working; one of the most fundamental properties is the Formula mass of their compounds.

In this experiment the Formula mass of a low boiling-point liquid is determined by the **Dumas Method** (John Dumas, 1800-1884). The procedure involves vaporising the liquid into a fixed-volume vessel at a measured temperature and barometric pressure. From the data and the use of ideal gas law equation (assuming ideal gas behaviour) the moles of vaporised liquid, n_{vapor} , is calculated:

$$N_{\text{vapor}} = \frac{P.V}{RT} = \frac{\text{Pr essure}(atm)xVolume(L)}{R(L.atm / molK)xTemperature(K)}$$

In this equation, R is universal gas constant equal to 0.08206 L.atm/mol.K, P is the barometric pressure, V is the volume of the vessel (L) into which the liquid is vaporized, and the T is the temperature of the vapour.

The mass of vapor, m_{vapor} , is determined from the difference between the empty vessel and the vapor-filled vessel.

$$m_{\text{vapor}} = m_{\text{(flask+vapour)}} - m_{\text{flask}}$$

The formula mass of the compound, FM_{compound}, is then calculated from data:

FM compound =
$$\frac{m_{vapor}}{n_{vapor}}$$

Gases and liquids that have relatively large intermolecular forces and large molecular volumes do not calculate according to the ideal gas law equation; in fact, some compounds that we normally consider as liquids deviate significantly from ideal gas behaviour at temperatures at or slightly above their boiling points. At these conditions, the intermolecular force and molecular volume correction can be considered in the determination of the formula mass of compound by using a modification of the ideal gas law equation; this is called Van der Waal's equation.

$$\left(P + \frac{n^2 a}{V^2}\right)(V - nb) = nRT$$

In this equation, P, V, T, R and n have the same meaning as in first equation. A is an experimental value that is representative of the intermolecular forces of the vapour and b is an experimental value that is representative of the volume of the molecules.

For a more accurate determination of moles of vapour in the flask, Van der Waal's equation can be used instead of the ideal gas law equation. Some values of **a** and **b** for a number of low boiling-point liquids are listed in Table 1.

Table1. Van der Waal's constants for some low boiling-point compounds

Name	a	b
methanol	9.523	0.06702
ethanol	12.02	0.08407
acetone	13.91	0.0994
propanol	14.92	0.1019
hexane	24.39	0.1735
cyclohexane	22.81	0.1424
n-pentane	19.01	0.1460

PROCEDURE

You are to complete three trials in determining the formula mass of your unknown low boiling-point liquid.

- 1. Fit a clean, 125 mL Erlenmeyer flask with a one-hole stopper.
- **2.** Dry the flask either in a drying oven or by allowing it to air-dry.(*do not wipe dry or heat over a direct flame*)
- **3.** Weigh the dry flask and the one-hole stopper.
- **4.** Add about 6 mL of the unknown liquid to the flask and insert the stopper. You do not need to weigh the liquid and flask.
- **5.** Cover the stopper with a small piece of aluminium foil. Secure the aluminium foil with a rubber band and, with a pin, pierce the aluminium foil several times at the point where the foil covers the hole in the rubber stopper.
- **6.** Fill a 600 mL beaker half-way with water; support it on wire gauze and near the top, with a support ring. Bring the water to a boil.
- 7. Remove the heat from the boiling water bath. Lower the flask in the bath and secure it with a utility clamp. Be certain that neither the flask nor clamp touches the beaker wall. Adjust the water level high on the neck of the flask.
- **8.** Resume heating until the water boils. When the liquid in the flask or vapours from the holes in the aluminium foil are no longer visible, continue heating for another 5 minutes. Record the **temperature of the boiling water**.
- **9.** Remove the flask and allow it to cool to room temperature. Sometimes the remaining vapour in the flask condenses. That's OK.
- **10.** Dry the outside of the flask and **weigh it**, the rubber stopper and vapour. (Don't forget to remove the aluminium foil before weighing)
- **11.** Do it again and again. Repeat the experiment for trials 2 and 3.
- 12. Fill the empty 125-mL Erlenmeyer flask to the brim with water and insert the one-hole stopper. Remove the stopper and measure the volume of the flask by transferring the water to a 50 or 100 mL graduated cylinder. Record the **volume**.
- 13. Find the barometer in the laboratory. Read and record the atmospheric pressure.

This experimental procedure was taken from the booklet of Eskişehir Technical University, Faculty of Engineering, Department of Chemical Engineering, General Chemistry Laboratory.

Report Sheet

DETERMINATION OF MOLECULAR MASS OF VOLATILE LIQUIDS

Student's Name :			Date:
Group No :			
Unknown Number	<u>Trial 1</u>	Trial 2	<u>Trial 3</u>
1. Mass of dry flask and stopper (g			
2. Temperature of boiling water (°C, K)			
3. Mass of dry flask, stopper and vapor (g))		
4. Volume of 125 mL flask (L)			
++=			
5. Atmospheric pressure (atm)			
Calculations (Ideal gas equation)			
Unknown Number	<u>Trial 1</u>	Trial 2	Trial 3
1. Moles of vapor, n _{vapor} (mol)			
2. Mass of vapor, m _{vapor} (g)		•••••	
3. Formula mass of compound (g/mol)		•••••	
4. Average Formula mass			
5. Standard deviation of Formula mass			

Calculations (van der Waal's equation)

Ask your instructor for the name of your unknown liquid. Using Van der Waal's equation and values of **a** and **b** for your compound, determine a more accurate Formula mass of the compound.

Unknown Number	<u>Trial 1</u>	<u>Tria</u>	<u>12</u> <u>Trial 3</u>
1. Moles of vapor, n _{vapor} (mol)			
2. Mass of vapor, m _{vapor} (g)			
3. Formula mass of compound (g/mol)			
4. Average Formula mass			

QUESTIONS

- 1. If the outside of the flask is not dried after vaporizing liquid, will the formula mass of the compound be too high or too low? Explain.
- 2. Suppose you assumed that the atmospheric pressure was one atmosphere instead of the pressure that you recorded. Would this have caused you to report a formula mass that was too high or too low?
- **3.** If all of the unknown liquid does not vaporize in the 125 mL flask, will the reported formula mass be too high or too low? Explain.

THERMAL DECOMPOSITION OF POTASSIUM CHLORATE AND DETERMINATION OF MOLAR VOLUME OF OXYGEN

MATERIALS AND REAGENTS

- KCIO₃ mixture
- O₂ gas
- Distilled water

CAUTIONS

- This experiment may be dangerous if performed improperly. Before you begin, read carefully:
- a. Wear safety glasses
- **b.** Use a clean 200-mm Pyrex test tube; there must be no evidence of black residue in the test tube. Do not dry the inner wall of the test tube with a paper towel.
- **c.** Your instructor must approve your apparatus before heating the unknown KCIO₃ mixture in Part A.

PURPOSE

To determine the percent potassium chlorate in a heterogeneous mixture To determine the molar volume of oxygen gas at 273 K and 760 torr

INTRODUCTION AND THEORY

The molar volume of a gas is the volume one mole (6.023x10²³ atoms or molecules) of gas occupies at a specified temperature and pressure. At standard temperature and pressure, (STP) one mole of an ideal gas occupies 22.4 liters that is its molar volume is 22.4 liters at STP.

In addition to determining the molar volume of oxygen gas (a real gas) in this experiment the percent by mass of potassium chlorate KCIO₃ in a heterogeneous mixture is determined. When potassium chlorate is heated it decomposes to potassium chloride KCI and oxygen gas¹ according to the equation:

$$2 \text{ KCIO}_3 \text{ (s)} -\Delta \rightarrow 2 \text{KCI}(\text{s}) + 3 \text{O}_2 \text{ (g)}$$

$$(6.1)$$

 $^{^1}$ KCIO3 decomposes slowly at 400 $^{\rm o}C$ but with a catalyst such as MnO2 or $\,Fe_2O_3$ it readily decomposes nearly to $\,$ completion at about 270 $^{\rm o}C$

To make these two determinations in the experiment two important measurements are made:

- (1) The O₂ gas given off from the decomposition is collected and its volume is measured and
- (2) the mass difference of the KCIO₃ mixture before and after heating is measured.

The mass loss of the KCIO₃ mixture is due to the mass of O_2 gas evolved in the reaction. The volume of O_2 gas collected under the temperature and pressure conditions of the experiment is adjusted to STP conditions and the mass of O_2 is converted to moles; knowing its number of moles and its volume at STP the molar volume of O_2 gas can be calculated.

$$\frac{Vo_{2(STP)}}{nO_2} = \text{molar volume of O}_2$$
 (6.2)

Volume of Collected Oxygen at STP

In this experiment the evolved O_2 gas is collected by displacing an equal volume of water since the O_2 gas is bubbled through the water it is considered "wet" meaning that the O_2 gas is assumed to be saturated with water vapor at the temperature of collection. Therefore the pressure exerted by this volume of "wet" O_2 is due to the O_2 gas and the water vapor. The pressure of the "dry" O_2 only is calculated, using Dalton's Law of partial pressures.

$$P_T = PO_2 + PH_2O$$
; for oxygen gas: $po_2 = P_T - PH_2O$ (6.3)

The vapor pressure of the water vapor pH_2O at the gas collecting temperature (obtained from Appendix E) is subtracted from the total pressure of the gasses P_T in the gas collecting flask. Experimentally the total pressure of the gaseous mixture is adjusted to that of atmospheric pressure by using a leveling tank. Atmospheric pressure is read from the laboratory barometer.

Once the experimental values for the volume pressure and temperature of the O_2 gas are determined the volume of the O_2 gas at STP (273K and 760 torr) conditions is calculated using a combination of Boyle's Law (P α 1/V) Charles Law (V α T).

$$V_{0_{2}}(\text{at STP}) = VO_{2} \text{ exp't x } \frac{PO_{2} \text{ exp't}(torr)}{760torr} \text{ x } \frac{273K}{TO_{2} \text{ exp't}(K)}$$

$$Boyle'sLaw \qquad Charles'Law$$

$$correction \qquad correction$$
(6.4)

This value is used in (Equation 6.2)

Percent KCIO₃ in Mixture

Once the moles of O₂ gas evolved in the reaction is known, the moles and mass of KCIO₃ in the mixture can be calculated. The percent KCIO₃ in the mixture can be calculated. The percent KCIO₃ in the mixture is calculated by dividing this mass of KCIO₃ by that of the original mixture and multiplying by 100.

$$\frac{mass\ of\ KCIO_3}{mass\ of\ mixure} \times 100 = \% KCIO_3$$
 (6.5)

PROCEDURE

Two trials are required in this experiment. To hasten the analyses, prepare two samples in Part A and perform the experiment with a partner-a partner you can trust!

A. Sample Preparation and Setup of Apparatus

- **1. The Mass of Sample.** In a clean, dry, previously-weighed ($\pm 0,001$ g) 200-mm test tube, weigh a maximum of 0.2 g of a KCIO₃ mixture contains KCIO₃, a catalyst, and a non-volatile impurity.
- **2. Setup of the 'Reaction' Test Tube.** Assemble the apparatus shown in figure 6.1. Clamp the Pyrex test tube to the ring stand at an angle of at least 45° from the horizontal and connect the gas delivery tube (rubber or Tygon tubing) to the pneumatic trough. Fill the trough about 2/3 full with water. Connect the gas delivery tube to the Pyrex tube containg the sample.
- **3. Setup of the 'Gas-Collecting' Test Tube.** Fill the 200-mm gas collecting test tube with water to the rim, cover with a piece, filter paper,² and invert it³ in the pneumatic trough (Figure 6.2)⁴, but not over the gas outlet. Remove the filter paper. Support the test tube with

² A glass plate may be substituted for the filter paper

³ No air bubbles should appear in the inverted test tube

⁴ Consult your instructor on this technique.

a ring stand and clamp. Make all connections air-light and make sure no bubbles enter the gas-collecting test tube. Obtain your instructor's approval before continuing.

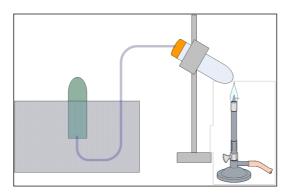


Fig. 1. Experimental Setup

B. Collection of the Oxygen Gas

Caution: Once you being Part B.1, you must proceed through Part B.3 without interruption. Therefore, read this section carefully before proceeding.

- 1. Expel the Heated Air. Heat the 'reaction' test tube above the sample. After the heated air is not longer expelled (bubbles of air do not appear at the gas outlet in the pneumatic trough) and while continuing to apply heat to the upper portion of the gas outlet in the pneumatic trough. Assistance from your partner is required. Proceed to Part B.2-do not stop!
- 2. Generate and Collect the Oxygen Gas. Move the Bunsen flame to the KCIO₃ mixture. Gently heat the mixture at first; then more strongly as the evolution rate of O₂ gas decreases. Heat all sides of the Pyrex test tube until no more gas is evolved (Caution: do not let molten KCIO₃ or the burner flame come in contact with the rubber stopper.) Proceed to Part B.3-do not stop!

3. When No More Gas Is Evolved. When there is no further generation of O₂ gas, first, disconnect the gas delivery tube from the 'reaction' test tube, and second, remove the heat, and then allow to cool. Your partner's assistance is required here also.⁵

C. Determination of the Temperature, Volume, and pressure of the Oxygen Gas

- **1. Temperature Determination.** Record the temperature of the water in the pneumatic trough.
- **2. Pressure Determination.** Adjust the pressure inside the gas-collecting test tube to atmospheric pressure by raising or lowering it until the water levels inside and out are the same (Figure 6.3). You may need to transfer it to the leveling tank in the laboratory. Ask your instructor for advice on equalizing the O₂ gas pressure in the leveling tank.

Read and record the barometric pressure in the laboratory.

3. Volume Determination. Place a piece of filter paper under the mouth of the test tube (from Part C.2), remove the test tube from the water, and place it right side up in your test tube rack.

Dry the outside of this test tube and weight (± 0.001 g) it and the remaining water (without the filter paper). Now, fill the test tube to the brim with water, slide a piece of filter paper over the rim, remove it, and again weight the test tube and its contents, now full with water. Assuming the density of water to be 1.00g/cm³, the mass difference, in grams, equals the volume of water displaced, in cm³, and the volume of the oxygen gas evolved in the reaction.

D. Mass of Oxygen Evolved

1. Determination of a Mass Difference. Weigh (10.001g) the cool 200-mm "reaction" test tube. Compare this mass to that in Part A.1.

⁵ If the reaction test tube cools before the gas delivery tube is disconnected, water from the pneumatic through will be drawn back into the hot salt mixture, causing the test tube to break. Also, an error in the volume of evolved oxygen occurs. If this does accidentally happen, you will need to repeat the trial.

This experimental procedure was taken from the booklet of Eskişehir Technical University, Faculty of Engineering, Department of Chemical Engineering, General Chemistry Laboratory.

Report Sheet

THERMAL DECOMPOSITION OF POTASSIUM CHLORATE AND DETERMINATION OF MOLAR VOLUME OF OXYGEN

Student's Name :		Date:
Group No :		
Unknown Sample No.	Trial1	Trial2
1. Mass of 'reaction' test tube + KCIO ₃ mixtur	e (g)	
2. Mass of 'reaction' test tube (g)		
3. Mass of KCIO ₃ mixture (g)		
4. Instructor's approval of apparatus		
5. Temperature of water (°C)		
6. Vapor pressure of H_2O at ${}^{o}C$		
(Appendix E) (torr)		
7. Barometric Pressure (torr)		
8. Mass of 'gas-collecting' test		
tube and water (full) (g)		
9. Mass of 'gas-collecting' test tube		
and water (partial) (g)		
10. Volume of water displaced		
(density of H ₂ O is 1.00g/cm ³)(L)		•••••
11. Volume of O ₂ gas collected (L)		
12. Mass of 'reaction' test tube and contents		
after heating (g)		
Molar Volume of O_2 Gas		
1. Pressure of 'dry' O ₂ gas (torr)		
2. Volume of O ₂ gas at STP (L)		
3. Mass of O ₂ gas produced (g)	•••••	••••

4.	Moles of O ₂ gas evolved (mol)						
5.	Molar volume of O ₂ gas at STP (L/n	nol)					
6.	Average molar volume of O2 gas at	STP (L/1	mol) .				
]	Percent KCIO ₃ in Mixture						
	1. Moles of KCIO ₃ in mixture (mol)		••••			• • • • • • • • • • • • • • • • • • • •	• • • • • •
	2. Mass of KCIO ₃ in mixture (g)						
	3. Mass of original mixture (g)						
	4. Percent of KCIO ₃ in mixture (%)						
	5. Average percent of KCIO ₃ in mix	ture (%)	••••				
					T		T
	Class Data/Group % KCIO ₃ in the salt mixture	<u>1</u>	<u>2</u>	<u>3</u>	4	<u>5</u>	<u>6</u>
	Sample no. ()						

Determine the mean value for the collected data, the average deviation and the standard deviation.

QUESTIONS

1. O_2 gas is slightly soluble in water. How does this affect the reported moles of KClO₃ decomposed in the mixture? Explain.

2.

- **a.** When the water level inside the gas collecting test tube is higher than it is outside, is the O_2 gas pressure greater or less than atmospheric pressure? Explain.
- **b.** When you equilibrate the gas pressures in Question 2a, does the volume of O_2 gas increase or decrease? Explain.
- c. Suppose you did not equilibrate the gas pressure to atmospheric pressure, but assumed the pressure of the O₂ gas equals atmospheric pressure. Are the reported moles of evolved O₂ gas greater or less than actual? Explain.
- **3.** If an air bubble accidentally enters the gas-collecting test tube, how would this affect:
 - a. The reported number of moles of KCIO₃ decomposed? Explain.
 - **b.** The *reported* molar volume of O_2 gas? Explain.
- **4.** In part B.1, if the 'reaction' test tube is not expelled prior to the generation of the oxygen gas from the decomposition reaction,
 - **a.** Will the calculated molar volume of O₂ gas be too high or too low? Explain.
 - **b.** Will the KCIO₃ percentage by affected? How? Explain.

INTERMOLECULAR INTERACTIONS: DISSOLUTION OF ALCOHOLS IN WATER AND ETHER

MATERIALS AND REAGENTS

- Isopropyl alcohol
- Ethylene glycol
- Glycerine
- Ether*
- Test tubes
- Dropper

*Caution: Ether is highly flammable and volatile substance.

PURPOSE

To examine the effect of intermolecular interactions on the solubility of substances.

INTRODUCTION AND THEORY

Solution is dispersion of solute molecules or ions in the solvent and is a single phase. Each of the substances in a solution is called a component of the solution. The material being dissolved is called the "solute", and the other component that dissolves the solute is the "solvent". Either or both components can be solid, liquid or gas. The greater the similarity between the molecules of solute and solvent, the easier a solution to form. Attractions between solute and solvent molecules help them disperse among one another. Usually solvent molecules surround the solute particle, forming a shell. Thus each solute molecule is said to be "solvated" (or, when the solvent is water, "hydrated"). For example, when an ionic compound dissolves in water, its ions are separated as a result of being attracted by the polar water molecules.

Solvents are classified as "polar" or "nonpolar" according to the molecular polarity in their molecules. A polar molecule has a dipole moment. The molecules nonpolar solvents have no dipole moments.

Nitrobenzene (C₆H₅NO₂), water (H₂O), ethyl alcohol (C₂H₅OH), phenol (C₆H₅OH), ammonia (NH₃), chloroform (CHCl₃) are polar solvents. Hexane (C₆H₁₄), benzene (C₆H₆), carbontetrachloride (CCl₄) are nonpolar solvents. Polar solvents will dissolve polar solutes or ionic solutes. Nonpolar solvents will dissolve nonpolar solutes. This rule is called "Like dissolves like".

PROCEDURE

- 1. Introduce 20 drops (1 mL) of water into one test tube and 20 drops of ether into another test tube.
- **2.** Add 10 drops of isopropyl alcohol to each solvent in the tubes. Shake them for a while, wait until the solutions clarify.
- **3.** Observe the solubility. When the dissolution does not occur between solvent and solute, two different phases form. Since the phases are colorless, a colored paper should be placed behind the tubes to see the phase separation.
- **4.** Apply the same procedure for other alcohols and report your observations in the table.

Reference: H. Güler, D. Saraydın, U. Ulusoy, Genel Kimya Laboratuvarı, Hatipoğlu yayınları, Ankara, 1993

<u>This experimental procedure was taken from the booklet of Eskişehir Technical University, Faculty of Science, Department of Chemistry, General Chemistry Laboratory.</u>

Report Sheet INTERMOLECULAR INTERACTIONS: DISSOLUTION OF ALCOHOLS IN WATER AND ETHER

Student's Name	:	Date:

Group No :

The alcohol	Structural formula	Solubility in water	Solubility in ether

QUESTIONS

Explain the solubilities of isopropyl alcohol, etylene glycol and glycerine in water and ether, considering the molecular structures of both solvent and solute.

COLLIGATIVE PROPERTIES: FREEZING POINT DEPRESSION AND BOILING POINT ELEVATION

MATERIALS AND REAGENTS

- Cyclohexane
- Unknown solute

CAUTIONS

Be careful while working with cyclohexane and dispose the waste of cyclohexane and cyclohexane solution in the "Waste Liquids" container.

PURPOSE

To determine the formula mass (molecular weight) of a nonvolatile, non-electrolyte solute by observing the difference between the freezing points of a solvent and a solution.

INTRODUCTION AND THEORY

The addition of a nonvolatile solute to a solvent produces characteristic changes in several physical properties of the solvent. It lowers the vapour pressure of the solvent, it increases the boiling point of the solvent, it decreases the freezing point of the solvent, and it creates an osmotic pressure for the solvent. These physical properties are called colligative properties because these changes depend only upon the number of solute particles dissolved in the solvent, not the kind of solute particles.

For example, one mole of glucose or urea (neither dissociate) lower the freezing point of one kilogram of water by 1.86 °C; whereas one mole of sodium chloride (it dissociates in solution, NaCl (aq) \rightarrow Na⁺(aq) + Cl⁻ (aq)) lowers the freezing point of one kilogram of water by nearly twice that amount (\approx 3.72 °C) because it provides twice as many moles of solute particles per mole of solute as do glucose or urea. When salt is used to freeze ice cream the salt-ice-water mixture used as a low temperature bath is at a lower temperature than an ice-water mixture alone. Antifreeze (ethylene glycol) added to the cooling system of the automobile reduces the probability of freeze-up in the winter and boiling in the summer

because the antifreeze-water solution has a lower freezing point and a higher boiling point than pure water.

These changes in the properties pure water, when it dissolves a non-volatile solute, are illustrated by the phase diagram in Figure 1, a plot of vapour pressure as a function of temperature. The solid lines refer to the equilibrium conditions between the respective phases for pure water; the second line represent the same conditions for an aqueous solution.

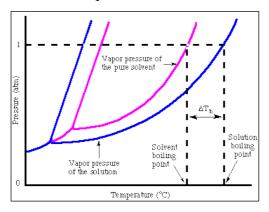


Figure 1. Phase diagram for water and an aqueous solution

When the vapour pressure of water is 760 torr, it boils at 100 °C. When a non-volatile solute is dissolved in water to form a solution, solute molecules occupy a part of the surface area. This, inhibits movement of some water molecules into the vapour state causing a vapour pressure lowering of the water (Figure 1), lower than 760 torr. Since the vapour pressure is less than 760 torr, the solution (more specifically, the water in the solution) no longer boils at 100 °C. For the solution to boil its vapour pressure must be increased to 760 torr; this is done by increasing the temperature above 100 °C. This is the boiling point elevation (Figure 1) of the water due to the presence of the solute.

A solute added to water also affects its freezing point. The normal freezing point of water is 0 °C, but in the presence of a solute, the temperature must be lowered (the energy of the water molecules must be lowered to increase the magnitude of the intermolecular forces, so that the water molecules "stick") below 0 °C before freezing occurs; this is the freezing point depression of the water due to the solute.

The changes in the freezing point, ΔT_f , and the boiling point, ΔT_b , are directly proportional to the molality of the solute in solution. The proportionality is a constant, characteristic of

the actual solvent. For water the freezing point constant, K_f, is 1.86 °C• kg/mol and the boiling point constant, Kb, is 0.512 °C• kg/mol.

$$\Delta T_f = |t_f, \text{ solvent} - t_f, \text{ solution}| = K_f m$$
 (1)

$$\Delta T_b = |t_b, \text{ solvent} - t_b, \text{ solution}| = K_b m$$
 (2)

t_f represents the freezing point and t_b represents the boiling point of the system.

 $\mid t_f$, solvent – t_f , solution \mid represents the absolute temperature difference in the freezing point change.

$$molality, m = \frac{mol\ solute}{kg\ solvent} = \frac{(grams\ /\ formula\ mass)}{kg\ solvent}$$
 (3)

K_f and K_b values for various solvents are listed in Table 1.

In this experiment the formula mass of a solid solute is determined, using cyclohexane as a solvent, by measuring the freezing point difference between pure cyclohexane and the solute/cyclohexane solution. From the $K_f(Table\ 1)$ and equations 1 and 3, you can calculate the moles of solute dissolved and from the measured mass, calculate formula mass of the solute.

The freezing points of the cyclohexane and the cyclohexane solution are obtained from a cooling curve-a plot of temperature vs. time (Figure 2). The cooling curve for pure cyclohexane reaches a plateau at its freezing point. The cooling curve for the solution does not reach a plateau, but continues to decrease slowly as the cyclohexane freezes. Its freezing point is determined at the intersection of two straight lines drawn through the data points above and below the freezing point (Figure 2).

Table 1. Molal Freezing Point and Boiling Point Constants for a Number of Solvents

Substance	Freezing	K _f	Boiling	K _b
	Point (°C)	(°C•kg/mol)	Point (°C)	(°C•kg/mol)
H ₂ O	0.0	1.86	100.0	0.512
Cyclohexane	-	20.0	80.7	2.69
Naphthalene	80.2	6.9	-	-
Camphor	179	39.7	-	-
Acetic acid	17	3.90	118.2	2.93
t-butanol	25.5	9.1	-	-

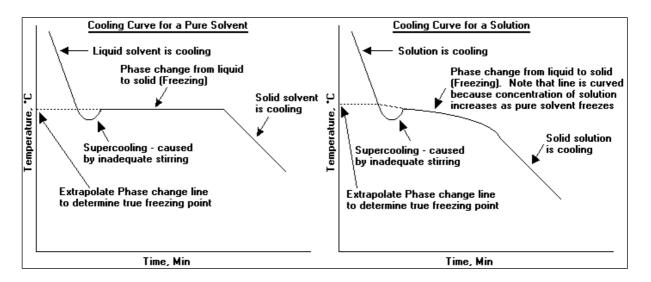


Figure 2. Cooling curves for a pure solvent and a solution

PROCEDURE

Obtain about 15 ml of cyclohexane. You'll use this throughout the experiment. Your laboratory instructor will issue to you about 1 gram of unknown solute. Record the unknown number on the Report Sheet.

A. Freezing Point of Cyclohexane (Solvent)

1. Construction of Apparatus. Assemble the apparatus shown in Figure 3. Use a 600-ml beaker as an outside, insulating beaker. You may want to place paper between the 600-ml beaker and the 400-ml beaker to better insulate the ice-water bath. Place about 300 ml of an ice-water slurry into the 400-ml beaker.

Obtain a thermometer clamp, mount it to the right stand, and position the thermometer into the test tube. Insert a wire stirrer into the test tube.

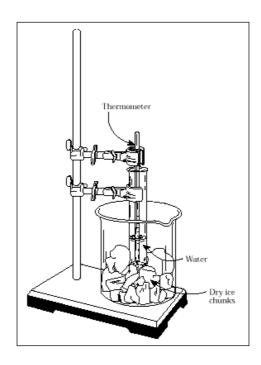


Figure 3. Freezing point apparatus

- **2. Preparation of Cyclohexane.** Weigh $(\pm 0.01 \text{ g})$ your clean, dry 200 mm-test tube in a 250-ml beaker. Add approximately 10 g of cyclohexane to the test tube and reweigh. Record these two masses on the Report Sheet. Place the test tube and cyclohexane in the ice-water bath (Figure 3). Secure the test tube with a utility clamp. Insert the thermometer and wire stirrer into the test tube. Make sure the thermometer extends down into the cyclohexane.
- 3. Data for the Freezing Point of Cyclohexane. While stir,ing with the wire stirrer, record the temperature ($\pm 0.1 \, \text{C}^{\circ}$) readings at timed intervals (30 or 60 seconds) on the Report Sheet. The temperature remains virtually constant at the freezing point until the solidification is complete. Continue recording until the temperature begins to drop again.
- **4. Plot the Data.** On linear graph paper, plot the temperature (°C, vertical axis) vs. time (sec, horizontal axis) to obtain the cooling curve for cyclohexane. Have your instructor approve your graph.

B. Freezing Point of Cyclohexane + Unknown Solute

Three freezing point trials for the solution are to be completed. Successive amounts of unknown sample are added to the cyclohexane in Parts B.4 and B.5.

- **1. Mass of Solid Solute.** Dry the outside of the test tube containing the cyclohexane and reweigh (± 0.01 g). Weigh approximately 0.1 to 0.3 g of unknown solute (ask your instructor for the approximate mass to use) on weighing paper. Quantitatively transfer the unknown solid solute to the cyclohexane in the 200-mm test tube.⁶
- 2. Data for the Freezing Point of Solution. Determine the freez,ing point of this solution in the same way as that of the solvent (Part A.3). When the solution nears the freez,ing point of the pure cyclohexane, record the temperature at more frequent time intervals (≈ 15 seconds). A break in the curve occurs as the freezing begis, although it may not be as sharp at that for the pure cyclohexane.
- **3. Plot the Data on the Same Graph.** Plot the temperature vs. time data on the same graph as that for the pure cyclohexane (Part A.4). Draw straight lines through the data points above and below the freezing point, the intersection of the two straight lines is the freezing point of the solution.
- **4. Repeat with Additional Solute.** Remove the test tube/solution from the ice-water bath. Add an additional 0.1 to 0.2 g of unknown solid solute, using the same procedure as in Part B.1. Repeat the freezing point determination and plot the temperature vs. time data on, again, the same graph (Parts B.2 and B.3).
- **5. Again, Repeat with Additional Solute.** Repeat Part B.4 with a n additional 0.1 to 0.2 g of unknown solid solute, using the same procedure as in Part B.1. Repeat the freezing point determination and again plot the temperature vs. time data on the same graph (Parts B.2 and B.3). You now should have four plots on the same graph.
- **6. Instructor's Approval.** Have your instructor approve the three temperature vs. time graphs that have been added to your first temperature vs. time graph for the pure cyclohexane.

DISPOSAL INFORMATION: Dispose of the waste cyclohexane and cyclohexane solution in the "Waste Liquids (Cyclohexane)" container.

⁶ In the transfer be certain that none of the solid solute adheres to the test tube wall. If some does, roll the test tube until the solute dissolves.

Report Sheet

COLLIGATIVE PROPERTIES: FREEZING POINT DEPRESSION AND BOILING POINT ELEVATION

Student's Name :	Date:
Group No :	
A. Freezing Point Of Cyclohexane (Solvent)
1. Mass of test tube + cyclohexane (g	<u> </u>
2. Mass of test tube (g)	
3. Mass of cyclohexane (g)	
4. Freezing point, from cooling curve	e (°C)
5. Instructor's Approval of graph	
A Engaring Daint Of Cyclob arong	Halmaran Caluta
A. Freezing Point Of Cyclohexane	- Unknown Solute
Unknown Solute No.	
	Trial 1 Trial 2 Trial 3 (Parts B1,B3) (Parts B.4) (Parts B.5)
1. Mass of test tube + cyclohexane (g	<u> </u>
2. Mass of cyclohexane (g)	
3. Mass of solute (g)	
4. Freezing point, cooling curve (°C)	
5. Instructor's Approval of graph	
Calculations	
1. K _f for cyclohexane	20.0 °C • kg /mol
2. Freezing point change, $\Delta T_f(^{\circ}C)$	
3. Mass of solute in solution, total (g	
4. Mass of cyclohexane in solution (l	<u>—————————————————————————————————————</u>
5. Moles of solute (mol)	*
6. Formula mass of solute	
7. Average formula mass of solute	

^{*}Show work for this calculation.

A. Cycl	ohexane	B. Cyclohexane + Unknown Solute					
		Trial 1		Trial 2		Trial 3	
Temp.	Time	Time	Temp	Time	Temp	Time	Temp
1							

Continue recording data on your own paper and submit it with the report sheet.

QUESTIONS

1. If the freezing point of the solution is erroneously read 0.2 °C lower than it should be, will the calculated formula mass of the solute be too high or too low? Explain.
 2. How will the freezing point change in this experiment be affected by a. The presence of a volatile solute? Explain. b. Two solutes that react according to the equation A + B → C? Explain.
3. If a thermometer is miscalibrated to read 0.5 °C higher that the actual temperature over its entire scale, will the reported formula mass of the solute be too high or too low?
4. If some solute adheres to the test tube wall in Part B.1, will the freezing point change be greater than or less than it should be? Explain.
5. If the cyclohexane is initially, but unknowingly, contaminated with a non-reactive, non-volatile solute, how (if at all) does this affect the reported formula mass of the solute? Why?
6. Explain why the freezing point of a pure solvent remains constant whereas the freezing point of a solution continues to decrease with time (Figure 2).

TITRATION OF WEAK ACIDS, pH and BUFFERS

PART 1: TITRATION OF WEAK ACIDS

REAGENTS and MATERIALS

- Buret
- Erlenmeyer flask
- Two brands of vinegar sample
- Standardized NaOH solution
- Phenolpthalein indicator

CAUTIONS

- Handle each of the solutions with care.
- Read the Lab Safety section for instructions in handling acids and bases.
- Do not smell non of the solutions, they are all irritants.

PURPOSE

To determine the percent by mass of acetic acid in vinegar sample.

INTRODUCTION AND THEORY

Household vinegar is a 4 to 5 percent (by mass) acetic acid, CH3COOh, solution. Generallly, caramel flavoring and coloring are also added to make the product sell better.

The percent by mass of acetic acid in vinegar is determined by titrating a measured mass of vinegar to a phenolphthalein end point with a measured volume of the standardized sodium hydroxide solution. The moles of acetic acid are calculated from the balanced equation

$$CH_3COOH(aq) + NaOH(aq) \rightarrow CH_3COO^-Na^+(aq) + H_2O$$

Since the volume and molarity of the standardized NaOH solution are known, the moles of NaOH used for the analysis are also known.

At the equivalence point in the titration, the number of moles of NaOH equals the number of moles of CH₃COOH. The mass of CH₃COOH in the vinegar is determined by the following equations:

$$M_{\text{ CH3COOH}} \times V_{\text{ CH3COOH}} = M_{\text{ NaOH}} \times V_{\text{ NaOH}}$$

$$mol \ CH_{3}COOH = M_{\text{ NaOH}} \times V_{\text{ NaOH}}$$

$$grams \ of \ CH_{3}COOH = mol \ CH_{3}COOH \times \frac{MW_{\text{ }} CH_{3}COOH}{mol \ CH_{3}COOH}$$

Finally the precent by mass of CH₃COOH in vinegar is calculated

$$\frac{grams \ of \ CH_3COOH}{grams \ of \ vinegar} x \ 100 = \% \ by \ mass \ of \ CH_3COOH$$

Titration set up was given in the figure.

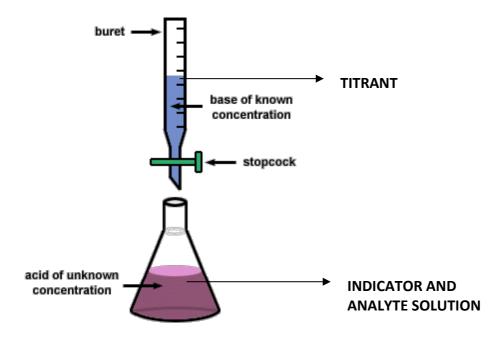


Figure: Experimental set-up for the titration procedure

PROCEDURE

Two samples of two vinegars are to be analyzed. At the beginning of the laboratory period, obtain 10 mL of each vinegar in separate 10 mL graduated cylinders.

Clean at least two 125 mL or 250 mL erlenmeyer flasks. When dry, label each flask and weigh.

1. Preparation of the vinegar sample:

Add about 5 mL of one of the brands of vinegar to clean, previously-weighed flask. Reweigh the flsak and the contents. Add 2 drops of phenolphthalein to the solution and rinse the wall of the flask with 20 mL of distilled water.

2. Prepare the Buret and Titration Setup:

Rinse a clean 50 mL buret with water first then with standardized NaOH solution. Fill the buret with standardized NaOH solution, eliminate air bubbles in the buret tip, read and record the initial volume. Place a white white sheet of paper under the flask containing the vinegar sample.

3. Titration:

Slowly add the NaOH saolution from the buret to the acid, swirling the flask after each addition. Occasionally rinse the wall of the flask with water by using wash bottle. Continue addition of the titrant until end point is reached. The end point in the titration should be within one half drop of a slight pink color. The color should stay for 30 seconds. After 30 seconds read and record the final volume of NaOH in the buret.

4. Repeat the titration with the same and another vinager.

This experimental procedure was taken from the booklet of Eskişehir Technical University, Faculty of Engineering, Department of Chemical Engineering, General Chemistry Laboratory.

Report Sheet 1 TITRATION OF WEAK ACIDS

Student's Name :	Date:
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Group No :

	Vinegar sample (1)		Vinegar	sample (2)
	Trial 1	Trial 2	Trial 1	Trial 2
1) Mass of flask (g)				
2) Mass of flask and vinegar (g)				
3) Mass of vinegar				
4) Final buret reading(mL)				
5) Initial buret reading(mL)				
6) Volume of NaOH used (mL)				
7) Molarity of NaOH				
8) Moles of NaOH added				
9) Moles of CH ₃ COOH in vinegar				
10) Mass of CH ₃ COOH in vinegar				
11) Percent by mass of CH ₃ COOH in vinegar				
12) Average percent by mass of CH ₃ COOH in vinegar				

QUESTIONS

- 1. A drop of NaOH solution adheres to the side of the flask and is not washed into the vinegar with the wash bottle. Does it cause any error in percent acetic acid in vinegar? Comment on this.
- 2. Vinegar samples are weighed rather than volume of it measured. Why?
- **3.** If you measure the volume of the vinegar sample instead of weigh, what additional data you need to find the percent by mass of CH₃COOH in the sample?
- **4.** If too much indicator (mililiters of it) is added to the vinegar in the flask, will the percent by mass of acetic acid be too high or low? Explain.

PART 2: pH and BUFFERS

REAGENTS and MATERIALS

- Mg strips
- 3 M H₂SO₄, 5M H₂SO₄, conc. H₃PO₄and 6M HC₂H₃O₂ solutions
- NaI, NaCl, NaNO₃, NH₄Cl, Na₂CO₃, CaO
- 1M NaOH

CAUTIONS

- Handle each of the solutions with care.
- Read the Lab Safety section for instructions in handling acids and bases.
- Do not smell non of the solutions, they are all irritants.

PURPOSE

To measure the pH of acids and bases

To determine the effectiveness of a buffer system

To determine the percent by mass of acetic acid

INTRODUCTION AND THEORY

pH Concept

The acidity or basicity of the most aqueous solutions arise due to low concentrations of hydrogen (H⁺) or hydroxide (OH⁻)ions. To express the acidity pH scale was introduced which can be expressed as negative logarithm of the hydrogen ion(H⁺).

pH=-log [H⁺] **or pH=-log [H**₃**O**⁺] where H₃O+ is called hydronium ion (both H⁺ and H₃O⁺ synonyms)

If the pH is greater than 7, solution is basic, if the pH is smaller than 7 it is so called acidic. Acidic solutions have a sour taste, cause a pricking sensation on the skin, and turn blue litmus red. Basic solutions have a bitter taste, is slippery to touch and turns red litmus blue.

Acids and bases can be divided into two as strong and weak. Stong acids and strong bases dissociate completely in water while weak acids and weak bases not.

Hydrogen ions (or hydronium ions) are produced by the hydrolysis of acids, and hydroxide ions are produced by the hydrolysis of bases.

$$CH_3COOH + H_2O \leftrightarrow CH_3COO^- + H_3O^+$$
 acid
 $NH_3 + H_2O \leftrightarrow NH_4^+ OH^-$ base

Water dissociates very slightly producing equal concentrations of hydronium and hydroxide ions. This process is called autoionization of water.

$$H_2O + H_2O \leftrightarrow H_3O^+ + OH^-$$

Ion products of hydronium and hydoxide in any aqueous solution can be described as

$$[H_3O^+]$$
 x $[OH^-]$ = $K_{W=}$ 1.0 x 10⁻¹⁴

At 25°C, the molar concentration of hydronium and hydroxide ions are equal $(1.0x10^{-7})$ and the pH is 7 (neutral).

Neutralization

When acids and bases react with each other a neutralization reaction occur.

For instance reaction of a strong base with stong acid produces equivalent amounts of salt and water.

$$NaOH + HC1 \rightarrow NaC1 + H_2O$$

How to measure the pH of a solution?

Exact pH of a solution is measured by a pH meter. On the other hand pH papers can also be used to make a rough pH measurement. Litmus paper is can also used to test whether a solution is acidic or bacis. Litmus paper turns into red while soaked into an acidic solution while basic solutions turns the litmus paper blue.

Buffers

In many areas of research, chemist want to have a solution that resists the addition of small amounts of acids and bases. An aqueous solution that performs this function is called a **buffer solution**. Logically, to resist the pH change via addition of acid and bas, a solution

must have an acidic component that will react with added base and neutralize it, and a basic component that will react with added acid. So, basically a buffer consists of a basic and acidic component together.

For instance acetic acid and acetate buffer system have an acidic component (acetic acid) and bacis component (acetate ion) together.

CH₃COOH + CH₃COO⁻ acetic acid-acetate buffer

CH₃COOH is a weak acid and CH₃COO⁻ is a conjugate base of CH₃COOH.

Now let us imagine what will happen, if few drops of HCl was added to this system.

Since HCl is an acid, it will react with tha basic component (CH₃COO⁻) and all the hydrogen ions coming from HCl is neutralized.

$$HCl+ CH_3COO^- \leftrightarrow CH_3COOH+ Cl-$$

Similarly, when a few drops of an base was added, it will be neutralized by the acidic component of the buffer system.

NaOH+ CH₃COOH↔ NaCH₃COO+H₂O

EXPERIMENTAL PROCEDURE

Part A. Acids and Acidic Solutions

A.1. Actions of acids on metals.

Polish small strips of Mg and place them separately in test tubes. Add 5 mL of 6M HCl to each metal. Write your observations on a table in the report sheet. Repeat the experiment with clean, rinsed metal strips with 3 M H₂SO₄, 5M H₂SO₄, and 6M HC₂H₃O₂. Note your observations.

A. 2. Effect of acid concentration on reaction rate

Place 5 ml of HCl into a test tube. Do the same with 0.1M HCl and 0.01 M HCl. Add a polished 1 cm strip of Mg each test tube and observe. Explain your observation.

A. 3. Oxidizing strengths of acids

Place about 0.2 g of NaI in each of two test tubes. Add several drops of conc. H₂SO₄(careful) to one of the test tubes and H₃PO₄ to other test tube. With moistened blue litmus paper, test any escaping gas from the test tubes. What color changes if any? What are the products of these reactions? How do sulfuric acid and phosphoric acid compare as oxidizing agents.

PART B. Bases and Basic Solutions

B.1. Reaction of Aqueous Sodium Hydroxide with Acid: Place about 3 mL of 1 M NaOH in a 50-mm test tube. Test the solution with litmus. Add drops of 6 M HCl - after each drop, agitate the mixture and test the solution with litmus, until a total of 1 mL (=20 drops) of HCl have been added. Account for your observation.

B.2. Repeat with Aqueous Sodium Carbonate: Repeat the above test, substituting 1 M Na₂CO₃ for the 1 M NaOH. Observe the solution closely while adding the HCl.

PART C. Acids and Bases

C.1. Neutralization of Base: Pipet 2 mL of 0,1 M HCl into a 150 mm test tube and add 1 drop of phenolphthalein indicator. Add drops of 0,5 M NaOH until a color change occurs (agitate the mixture after aech drop). Record the drops added.

C.2. Repeat the Neutralization with Other Acids: Repeat C.1, substituting 0,1 M H₂SO₄ and 0,1 M HC₂H₃O₂. Compare the acidity of the three acids.

C.3. pH of Acids and Bases: On the display table is a set of 8 labeled test tubes. Universal indicator has been added to each solution. Record the pH (a measure of the acidity of the solution) of each.

#1. 0.01 M HCl	#5. 0.01 M NaOH
#2. 0.01 M H ₂ SO ₄	#6. 0.01 M NH ₃
#3. 0.01 M HC ₂ H ₃ O ₂	#7. 0.01 M Na ₂ CO ₃
#4. 0.01 M H ₃ PO ₄	#8. 0.01 M Ba(OH) ₂

Comment on the acidity of solutions 1 through 4 and basicity of solutions 5 through 8. Notice that the molar concentrations of the acids and bases are the same.

This experimental procedure was taken from the booklet of Eskişehir Technical University, Faculty of Engineering, Department of Chemical Engineering, General Chemistry Laboratory.

Report Sheet 2 pH and BUFFERS

Student's Name	:			Date:
Group No	:			
A. Acids and Acid	ic Solutions			
1. Action of Acids	on Metals. In the follo	owing table, state	whether or	not a reaction is
observed. Indicate t	the relative reactivity (i.e	e., fast, moderate,	slow, etc.) o	f the metals with
the corresponding a	cid.			
	Reagents	Mg		
	6 M HCl			
	3 M H ₂ SO ₄			
	6 M HNO ₃			
	6 M HC ₂ H ₃ O ₂			
Explain Compare the	reaction rates with	the three	$HC_2H_3O_2$	concentrations
How are HCl and H	IC ₂ H ₃ O ₂ similar?			
How do HCl and H				
3. Oxidizing Stren	gth of Acids			
Observation with N	aI and H ₂ SO ₄			
Observation with N	aI and H ₃ PO ₄			

Compare the relative oxidizing strengths of H ₂ SO ₄ and H ₃ PO ₄
B. Bases and Basic Solutions
1. Reaction of Aqueous Sodium Hydroxide with Acid
Describe the progress of the reaction based upon your litmus tests
2. Repeat with Aqueous Sodium Carbonate
Describe the progress of the reaction based upon your litmus tests
C. pH of Acids and Bases:

Solution	pН	Solution	pН
0.01 M HCl		0.01 M NaOH	
0.01 M H ₂ SO ₄		0.01 M NH ₃	
0.01 M HC ₂ H ₃ O ₂		0.01 M Na ₂ CO ₃	
0.01 M H ₃ PO ₄		0.01 M Ba(OH) ₂	

INVESTIGATION OF FACTORS AFFECTING REACTION RATE

PURPOSE

The purpose of the experiment is to investigate the most important factors that affect the rate of a chemical reaction such as concentration of the reactants, temperature of the vessel, surface area of the reactants and presence of a catalyst

INTRODUCTION AND THEORY

Chemical kinetics is the study of chemical reaction rates, how reaction rates are controlled, and the pathway or mechanism by which a reaction proceeds from its reactants to its products.

Before a reaction can occur, the reactants must come into direct contact via collisions of the reacting particles. However, even then, the reacting particles (ions or molecules) must collide with sufficient energy, which is called "activation energy", to result in a reaction; if they do not, their collisions are ineffective and analogous to collisions of billiard balls. With these considerations in mind, we can qualitatively explain how the various factors influence the rates of reactions.

Reaction rates vary from the very fast, in which the reaction, such as the explosion of a hydrogen/oxygen mixture, is essentially complete in microseconds or even nanoseconds, to the very slow, in which the reaction, such as the setting of concrete, requires years to complete. The rate of a chemical reaction may be expressed as a change in the concentration of a reactant (or a product) as a function of time (e.g., per second)-the greater the change in the concentration per unit of time, the faster the rate of the reaction. Other parameters that can follow the change in concentration of a species as a function of time in a chemical reaction are color, temperature, pH, odor and conductivity. The parameter chosen for following the rate of a particular reaction depends on the nature of the reaction and the species of the reaction.

Let's examine now precisely what is meant by the expression rate of reaction. Consider the hypothetical reaction

$$A + B \longrightarrow C + D$$

The rate of this reaction may be measured by observing the rate of disappearance of either of the reactants A and B, or the rate of appearance of either of the products C and D. In practice, then, one measures the change of concentration with time of either A, B, C or D. Which species you choose to observe is a matter of convenience. For example, if A, B and D are colorless and C is colored, you could conveniently measure the rate of appearance of C by observing an increase in the intensity of the color of the solution as a function of time. Mathematically, the rate of reaction may be expressed as follows:

Rate of disappearance of A = Change in concentration of A =
$$\Delta$$
[A]
Time required for change Δ t

Rate of appearance of C = Change in concentration of C =
$$\Delta$$
[C]
Time required for change Δ t

In general, the rate of the reaction will depend upon the concentration of the reactants.

Thus, the rate of our hypothetical reaction may be expressed as

Rate =
$$k [A]^x [B]^y$$

where [A] and [B] are the molar concentrations of A and B, x and y are the powers to which the respective concentrations must be raised to describe the rate. k is the "specific rate constant". It should be emphasized that k has a definite value that is independent of the concentration. It is characteristic of a given reaction and depends only on temperature. Once the rate is known, the value k can be calculated.

A. Concentration

Changing the concentration of a solution alters the number of particles per unit volume. The more particles present in a given volume, the greater the probability of their colliding. Hence, increasing the concentration of a solution increases the number of collisions per unit time and therefore the rate of reaction.

In this experiment, the effect of concentration on the rate of a chemical reaction will be investigated with the addition reaction of formaldehyde (H₂CO) with bisulphide (HSO₃⁻) ion

$$H$$
 $C=O + HSO_3^ \longrightarrow$ H $C=OHSO_3^-$

Since the concentration of formaldehyde is higher than the concentration of bisulphide ions, all bisulphide ions will be ionically bonded to formaldehyde molecules. Formaldehyde reacts with the NaHSO₃ solutions of different concentrations. The time is measured for all HSO₃⁻ ions reacted with formaldeyde. A solution which contains Na₂SO₃ and phenolphthalein is used to determine the time that all HSO₃⁻ ions reacted with formaldehyde. The reaction is,

$$H$$
 $C=O + SO_3^{2-} + H_2O$ \longrightarrow H $C=OHSO_3^{-} + OH^{-}$

The hydroxyl ion occured in this reaction reacts quickly with HSO₃⁻ ion and forms SO₃²⁻ ion and water and SO₃²⁻ concentrate in solution remains constant.

$$OH^- + HSO_3^- \xrightarrow{fast} SO_3^{2-} + H_2O$$

Since the reaction is much faster than the addition reaction of HSO₃⁻ with SO₃²⁻ ions, OHions quickly vanishes. When all HSO₃⁻ ions are reacted with formaldehyde, the concentration of OH⁻ ions in the solution will quickly increases and phenolphthalein will make the solution pink.

The pink color of the solution demonstrates that the reaction to be completed. The reaction rate depends only the concentration of HSO₃⁻ ions and the reaction is first order.

Reaction rate =
$$-\frac{d[HSO_3^-]}{dt}$$
 = $k[HSO_3^-]$ mol L⁻¹ s⁻¹

MATERIALS AND REAGENTS

- Solution A: 0.3 M formaldehyde
- Solution B: (0.2 mole NaHSO₃ + 0.03 mole Na₂SO₃ + phenolphthalein)/L solution
- 400 Ml Beaker
- 50 Ml Beaker (3)
- 10 Ml Pipette (3)
- Test Tubes
- Time-Keeper
- Thermometer

PROCEDURE

- A. Place 300 mL of distilled water into a beaker. Measure the temperature of the water.
- **B.** Place 12 mL of solution A into the first beaker, 12 mL of solution B into the second beaker and 20 mL of distilled water into the third beaker.
- C. Place 3 mL of solution A into four test tubes (dry and clean).
- **D.** Place 3 mL of solution B into other four test tubes (dry and clean). Add 2 mL of distilled water into the second test tube, 4 mL of distilled water into the third test tube and 6 mL of distilled water into the fourth test tube.
- **E.** Put the eight test tubes into the water bath and wait until the temperatures of solutions reaches the temperature of water.
- **F.** Pour one of the test tubes which contains solution A into the first test tube which contains solution B and start time-keeper. Record the time until solution turns pink.
- **G.** Repeat the same procedure for second, third and fourth test tubes. Report Sheet

B. Temperature

Since temperature is a measure of the average kinetic energy, an increase in temperature increases the kinetic energy of the particles. An increase in kinetic energy increases the velocity of the particles and therefore the number of collisions between them in a given period of time. Thus, the rate of reaction increases. Also, an increase in kinetic energy results in a greater proportion of the collisions having the required energy for reaction.

In this experiment, you will investigate the effect of temperature on the reaction of potassium permanganate with oxalic acid at different temperatures. The reaction is,

 $2KMnO_4 + 3H_2SO_4 + 5H_2C_2O_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 10CO_2$ or only with ions,

$$2MnO_4^- + 16H^+ + 5C_2O_4^{2-}$$
 \longrightarrow $2Mn^{2+} + 8H_2O + 10CO_2$

Purple coloured MnO_4^- ion turns to colourless Mn^{2^+} ion at the end of the reaction.

MATERIALS AND REAGENTS:

- 0.0005 M KMnO₄
- 0.25 M H₂SO₄
- 0.0025 M H₂C₂O₄
- Test tubes
- 400 mL beaker
- Time-keeper
- Thermometer

PROCEDURE:

- **A.** Place 300 mL of distilled water into a beaker. Heat the water until temperature is 70 °C.
- **B.** Place 5 mL of 0.0005M KMnO₄ and 1 mL of 0.25 M H₂SO₄ into five test tubes (dry and clean).
- C. Place 9 mL of 0.0025 M H₂C₂O₄ into other five test tubes (dry and clean).
- **D.** Put the ten test tubes into the water bath and wait until the temperatures of solutions reaches the temperature of water.
- **E.** When water bath temperature has dropped to 65 °C, Pour one of the test tubes which contains H₂C₂O₄ into one first test tube which contains KMnO₄ and H₂SO₄ and start time-keeper. Record the time until solution becomes colourless.
- F. Repeat the same procedure for 55 °C, 45 °C, 35 °C and 25 °C.

C. Surface area of the reactants

In heterogeneous reactions, the reaction rate increases by the increasing surface area of the reactants. Surface area of the solids increases with decreasing particle size.

In this experiment, you will investigate the effect of surface area on reaction rate by using a piece of chalk and chalk dust.

MATERIALS AND REAGENTS

- Test tubes
- A piece of chalk
- Chalk dust
- Concentrated HCl solution

PROCEDURE

- A. Place a piece of chalk into a test tube.
- **B.** Place some chalk dust into another test tube.
- C. Add 10-20 mL of concentrated HCl into both test tubes.

D. Catalyst

Catalysts, in some cases, are believed to increase reaction rates by bringing particles into close just a position in the correct geometrical arrangement for reaction to occur. In other instances, catalysts offer an alternative route to the reaction, one that requires less energetic collisions between reactant particles. f less energy is required for a successful collision, a larger percentage of the collisions will have the requisite energy and the reaction will occur faster. In other words, the activation energy decreases. Actually, the catalyst may take an active part in the reaction, but at the end of the reaction, the catalyst can be recovered chemically unchanged.

In this experiment, you will investigate the effect of catalyst on the reaction of potassium permanganate with oxalic.

MATERIALS AND REAGENTS

- 0.0005 M KMnO₄
- 0.25 M H₂SO₄
- 0.0025 M H₂C₂O₄
- 0.25 M MnSO₄
- Test tubes
- Time-keeper

PROCEDURE

- **A.** Place 5 mL of 0.0005M KMnO₄ and 1 mL of 0.25 M H₂SO₄ into two test tubes (dry and clean).
- B. Place 9 mL of 0.0025 M H₂C₂O₄ into other two test tubes (dry and clean). Add 0.25 M of MnSO₄ into one of the tubes.
- C. Pour the test tubes which contains H₂C₂O₄ into one first test tube which contains KMnO₄ and H₂SO₄ and start time-keeper. Record the time until solution becomes colourless.
- **D.** Repeat the same procedure for the test tube which contains H₂C₂O4 and MnSO₄.

Reference: H. Güler, D. Saraydın, U. Ulusoy, Genel Kimya Laboratuvarı, Hatipoğlu yayınları, Ankara, 1993

<u>This experimental procedure was taken from the booklet of Eskişehir Technical University, Faculty of Science, Department of Chemistry, General Chemistry Laboratory.</u>

Report Sheet INVESTIGATION OF FACTORS AFFECTING REACTION RATE

Student's Name : Date:

Group No :

PART A.

Number of tube	V _A / mL	V _B / mL	V _{water} / mL	Time for the reaction
1	3	3	0	
2	3	3	2	
3	3	3	4	
4	3	3	6	

Results

1.

Number of tube	[HSO ₃ -]/ mole L-1	Reaction Rate/	k/ s ⁻¹
		mole L ⁻¹ s ⁻¹	
1			
2			
3			
4			

- 2. Draw the reaction rate graphic as a function of [HSO₃-].
- **3.** Compare the k values of the reactions and comment.

PART B.

Experimental Data

Temperature/ °C	Time for the reaction/ s
25	
35	
45	
55	

Results

- 1. Draw the time for reaction graphic as a function of temperature.
- 2. Calculate the factor for the increasing reaction rate by increasing temperature.

Temperature range /ºC	Factor
25-35	
35-45	
45-55	
55-65	
Average	

PART C.

Experimental Data and Results

Record your observations and comment.

PART D.

Experimental Data

Record your observations.

Results

- 1. Comment your observations.
- 2. Write the chemical reaction equations.

QUESTIONS

- 1) Explain what the reaction rate is and what factors increase the rate of a chemical reaction.
- 2) The reaction below is carried out at a constant temperature and a constant volume container.

$$Zn_{(k)} + 2HCl_{(aq)} \rightarrow ZnCl_{2(aq)} + H_{2(g)}$$

According to this;

- a) Addition of purified water
- b) Addition of concentrated HCl
- c) The addition of $H_{2(g)}$

Explain how the reaction rates change for the steps above.

CHEMICAL EQUILIBRIUM AND DETERMINATION OF EQUILIBRIUM CONSTANT

MATERIALS AND REAGENT

- Sodium thiocyanate (NaSCN)
- Ferric nitrate (Fe(NO₃)₃)
- Distilled water
- Ruler
- Test tubes
- Pipettes

PURPOSE

- To learn about chemical equilibrium.
- To determine the equilibrium constant of a chemical reaction experimentally.

INTRODUCTION AND THEORY

A state of chemical equilibrium exists when the rate of the forward reaction is equal to the rate of the reverse reaction. Once equilibrium has established itself, the amounts of products and reactants are constant. Furthermore, if one of the product or reactant concentrations can be measured, it can be used to determine the remaining concentrations and the equilibrium constant. For a general reaction, we denote this equilibrium as:

$$a A + b B \leftrightarrow c C + d D$$
 (Eq. 1)

The ratio of the product concentrations to reactant concentrations in the following form, at the equilibrium point, defines the equilibrium constant, K_c, for the system:

$$K_c = \frac{\left[C\right]^c \left[D\right]^d}{\left[A\right]^a \left[B\right]^b}$$
 (Eq. 2)

Large values of K_c mean products dominate at equilibrium, whereas small values imply a

predominance of reactants.

In this experiment you will determine the value of the equilibrium constant for the reaction (Eq. 3) between ferric ion (Fe³⁺) and thiocyanate ion (SCN⁻).

$$Fe^{3+} + SCN^{-} \leftrightarrow FeSCN^{2+}$$
 (Eq. 3)

For which the equilibrium condition is
$$K_c = \frac{\left[FeSCN^{2+}\right]}{\left[Fe^{3+}\right]SCN^{-}}$$

To find the value of K_c, it is necessary to determine the concentration of each species Fe³⁺, SCN⁻ and FeSCN²⁺ in the system at equilibrium. This will be done colorimetrically, taking advantage of the fact that FeSCN²⁺ is the 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. For instance, 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 the concentrations is simply the inverse of the ratio of the two depths (Eq. 4).

$$h_1C_1 = h_2C_2$$
 (Eq. 4)

It should be noted that this procedure permits only a comparison between concentrations. It does not give an absolute value of either one of the concentrations. To know the absolute values, it is necessary to compare unknown solutions with a standard of known concentration.

For colorimetric determination of FeSCN²⁺ concentration, you must have a standard solution in which the concentration of FeSCN²⁺ is known. Such a solution can be prepared by starting with a known concentration of SCN⁻ and adding such a large excess of Fe³⁺ that essentially all the SCN⁻ is converted to FeSCN²⁺. Under these conditions, you can assume that the final concentration of FeSCN²⁺ is equal to the initial concentration of SCN⁻. Moreover, the dilution of the reactants during the reaction must be considered. The equilibrium concentrations can be calculated as below:

$$\begin{split} [FeSCN^{2+}]_{equilibrium} &= [FeSCN^{2+}]_{standard} \ x \ (h_{standard}/h_{equilibrium}) \\ [Fe^{3+}]_{equilibrium} &= [Fe^{3+}]_{initial} - [FeSCN^{2+}]_{equilibrium} \\ [SCN^{-}]_{equilibrium} &= [SCN^{-}]_{initial} - [FeSCN^{2+}]_{equilibrium} \end{split}$$

PROCEDURE

- 1. Clean five test tubes with distilled water and let them dry.
- 2. To each of these test tubes add 5 mL of 0.002 M NaSCN.
- **3.** Add 5 mL of 0.20 M Fe(NO₃)₃ into one of the test tubes. This tube will serve as the standard.
- **4.** For the other test tubes proceed as follows: Mix the given amounts in table for each test tube.

Test tube	1	2	3	4
Fe(NO ₃) ₃	5 mL 0.08M	5 mL 0.032 M	5 mL 0.0128 M	5 mL 0.00512 M
NaSCN	5 mL 0.002 M	5 mL 0.002 M	5 mL 0.002 M	5 mL 0.002 M

5. Now the problem is to determine the concentration of FeSCN²⁺ in each test tube relative to the standard test tube. Compare the color intensity in Standard test tube with that in each 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. Look down through the solutions. If color intensities appear identical, measure the heights of the solutions in the two tubes being compared. If not, take Standard test tube and pour out some of the standard into a clean test tube (you may need to pour some back) until the color intensities appear identical. Do this comparison for all four tubes.

Reference: H. Güler, D. Saraydın, U. Ulusoy, Genel Kimya Laboratuvarı, Hatipoğlu yayınları, Ankara, 1993

<u>This experimental procedure was taken from the booklet of Eskişehir Technical University, Faculty of Science, Department of Chemistry, General Chemistry Laboratory.</u>

Report Sheet

CHEMICAL EQUILIBRIUM AND DETERMINATION OF EQUILIBRIUM CONSTANT

Student's Name : Date:
Group No :

Test tube	Heights of unknown liquid	Heights of standard
	(cm)	(cm)
1		
2		
3		
4		

Results

Test	Initial concentrations			Equilibrium		
tube						constant
	$[Fe^{3+}]$	[SCN ⁻]	FeSCN ²⁺	[Fe ³⁺]	[SCN-]	K _c
1						
2						
3						
4						

QUESTIONS

$2SO_2(g) + O_2(g) \leftrightarrows 2SO_3(g)$					
1. $O_2(g)$ is added to the system.	a. The equilibrium shifts to the right.				
2. SO ₃ (g)is removed from the system.	b. The equilibrium shifts to the left.				
$3. SO_2(g)$ is removed from the system.	c. There is no change in the equilibrium				
position					
4. Catalyst is added to the system.					

1) Match the change in the balance system below with the character of the appropriate answer.

REDOX REACTIONS: INVESTIGATION OF METAL ACTIVITIES

MATERIALS AND REAGENTS

• Aqueous solution of ZnSO₄ Metallic Zinc

• Aqueous solution of FeSO₄ Metallic Iron

• Aqueous solution of SnCl₂ Metallic Tin

• Aqueous solution of CuSO₄ Metallic Copper

Aqueous solution of Pb(NO₃)₂
 Metallic Lead

Aqueous solution of AgNO₃
 Test tubes

PURPOSE

To determine the activites of various metals with the help of reactions between them and some metal solutions.

INTRODUCTION AND THEORY

The replacement of one component in a compound by another describes the "replacement (or substitution)" reaction. This reaction has the general formula:

$$AB + C \rightarrow A + BC$$

Processes which involve oxidation (the loss of electrons or the gain of relative positive charge) and reduction (the gain of electrons or the loss relative positive charge) are typical of these reactions. Use of Table 1, the activity series of common metals enables chemists to predict which oxidation-reduction reactions are possible. A more active metal, or one higher in the table, is able to displace a less active metal, one listed lower in the table, from its aqueous salt solution. Thus aluminum metal displaces copper metal from an aqueous solution of copper(II) chloride; but copper metal will not displace aluminum from an aqueous solution of aluminum(III) chloride.

$$2Al(s) + 3CuCl_2(aq) \rightarrow 3Cu(s) + 2AlCl_3$$

 $Cu(s) + AlCl_3(aq) \rightarrow No Reaction$

(Note that Al is oxidized to Al³⁺ and Cu²⁺ is reduced to Cu.)

Hydrogen may be displaced from water by a very active metal. Alkali metals are particularly reactive with water, and the reaction of sodium with water often is exothermic enough to ignite the hydrogen gas released.

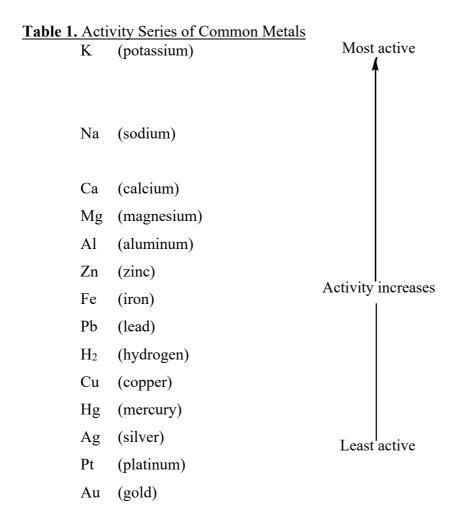
$$2Na(s) + 2H_2O(l) \rightarrow 2NaOH(aq) + H_2(g) + heat$$

(Note that Na is oxidized to Na⁺ and H⁺ is reduced to H₂.)

Active metals, those above hydrogen in the series, are capable of displacing hydrogen from aqueous mineral acids such as HCl or H₂SO₄ to release hydrogen gas, but copper will not.

$$Zn(s) + 2HCl(aq) \rightarrow ZnCl_2(aq) + H_2(g)$$

 $Cu(s) + H_2SO_4(aq) \rightarrow No reaction$



In this experiment, the relative activities of zinc, copper, iron, lead, tin metals will be determined by the reactions with their aqueous solutions.

PROCEDURE

- **A.** Place 5 mL of aqueous solutions of ZnSO₄, FeSO₄, SnCl₂, CuSO₄, Pb(NO₃)₂ and AgNO₃ in 6 test tubes respectively.
- **B.** Add a small piece of metallic zinc into the test tubes except the tube involves Zn²⁺ solution. Observe the reactions and report your observations.
- **C.** Apply the same procedure for other aqueous metal solutions and metals.

Reference: H. Güler, D. Saraydın, U. Ulusoy, Genel Kimya Laboratuvarı, Hatipoğlu yayınları, Ankara, 1993

This experimental procedure was taken from the booklet of Eskişehir Technical University, Faculty of Science, Department of Chemistry, General Chemistry Laboratory.

Report Sheet

REDOX REACTIONS: INVESTIGATION OF METAL ACTIVITIES

Student's Name	:	D	ate:
Group No	:		

Experimental Data

	The Metal Ion in the Aqueous Solution						
Metal	Zn ²⁺	Fe ²⁺	Sn ²⁺	Pb ²⁺	Cu ²⁺	$\mathbf{A}\mathbf{g}^{+}$	
Zn	-						
Fe		-					
Sn			-				
Pb				-			
Cu					-		

QUESTIONS:

1)	Define	the	fol	lowing	terms
1		uic	101	iow mg	wills.

Oxidation:

Reduction:

Activity:

Redox reaction:

2) Discuss the activities of the metals X, Y and Z in the reactions given below.

$$X + YSO_4 {\longrightarrow} XSO_4 {+} Y_2$$

$$Z + YSO_4 \rightarrow ZSO_4 + Y$$

$$Z + XSO_4 \rightarrow ZSO_4 + X$$