THE HASHEMITE UNIVERSITY

DEPARTMENT OF CHEMISTRY

LABORATORY MANUAL
FOR
EXPERIMENTAL CHEMISTRY FOR
ENGINEERING
CHEM. 110103108

Version 6

(2020)

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The Hashemite University

C h e m i s t r y Department

Chemistry Laboratory for Engineering Students (110103108)

Course Content :

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
1A	2A											3A	4A	5A	6A	7A	8A
1 **H** 1.008																	2 **He** 4.003
3 **Li** 6.941	4 **Be** 9.012											5 **B** 10.81	6 **C** 12.01	7 **N** 14.01	8 **O** 16.00	9 **F** 19.00	10 **Ne** 20.18
11 **Na** 22.99	12 **Mg** 24.31											13 **Al** 26.98	14 **Si** 28.09	15 **P** 30.97	16 **S** 32.07	17 **Cl** 35.45	18 **Ar** 39.95
19 **K** 39.10	20 **Ca** 40.08	21 **Sc** 44.96	22 **Ti** 47.88	23 **V** 50.94	24 **Cr** 52.00	25 **Mn** 54.94	26 **Fe** 55.85	27 **Co** 58.93	28 **Ni** 58.69	29 **Cu** 63.55	30 **Zn** 65.38	31 **Ga** 69.72	32 **Ge** 72.59	33 **As** 74.92	34 **Se** 78.96	35 **Br** 79.90	36 **Kr** 83.80
37 **Rb** 85.47	38 **Sr** 87.62	39 **Y** 88.91	40 **Zr** 91.22	41 **Nb** 92.91	42 **Mo** 95.94	43 **Tc** (98)	44 **Ru** 101.1	45 **Rh** 102.9	46 **Pd** 106.4	47 **Ag** 107.9	48 **Cd** 112.4	49 **In** 114.8	50 **Sn** 118.7	51 **Sb** 121.8	52 **Te** 127.6	53 **I** 126.9	54 **Xe** 131.3
55 **Cs** 132.9	56 **Ba** 137.3	57 **La*** 138.9	72 **Hf** 178.5	73 **Ta** 180.9	74 **W** 183.9	75 **Re** 186.2	76 **Os** 190.2	77 **Ir** 192.2	78 **Pt** 195.1	79 **Au** 197.0	80 **Hg** 200.6	81 **Tl** 204.4	82 **Pb** 207.2	83 **Bi** 209.0	84 **Po** (209)	85 **At** (210)	86 **Rn** (222)
87 **Fr** (223)	88 **Ra** 226	89 **Ac**† (227)															

	58 **Ce** 140.1	59 **Pr** 140.9	60 **Nd** 144.2	61 **Pm** (145)	62 **Sm** 150.4	63 **Eu** 152.0	64 **Gd** 157.3	65 **Tb** 158.9	66 **Dy** 162.5	67 **Ho** 164.9	68 **Er** 167.3	69 **Tm** 168.9	70 **Yb** 173.0	71 **Lu** 175.0
*Lanthanides														
†Actinides	90 **Th** 232.0	91 **Pa** (231)	92 **U** 238.0	93 **Np** (237)	94 **Pu** (244)	95 **Am** (243)	96 **Cm** (247)	97 **Bk** (247)	98 **Cf** (251)	99 **Es** (252)	100 **Fm** (257)	101 **Md** (258)	102 **No** (259)	103 **Lr** (260)

Experiment (1)

Safety and Equipments

Welcome to the Chemistry lab! Chemistry is an experimental science and you will learn many important things by doing experiments. In this first laboratory, we want you to get familiar with safety rules in the laboratory. Also, you will be introduced to the laboratory so that you'll get familiar with the lab and your equipment. **There is no grade for this first lab session!**

OBJECTIVES OF THE PRACTICAL COURSE IN CHEMISTRY

1. To learn basic laboratory skills with standard chemical equipment.

2. To carry out experiments involving laboratory chemicals in a safe and correct way with minimum waste.

3. To learn to observe and report the results of simple chemical experiments correctly.

4. To learn to explain observations using the concepts you have learned in the lecture course.

Read the following safety rules before going into the laboratory and abide by them.

Wear safety glasses at all times. Prescription glasses are adequate but contact lenses are not adequate.

1- Always wear your laboratory coat. Do not wear clothing that hinders free movement of your hands or hangs loose outside your laboratory coat.

2- Do not work in a laboratory if no lecturer or technician is present. Read the experimental instructions carefully before starting the work. Especially note any precautions that must be taken.

3- Never eat, drink, or smoke in the laboratory. Never taste chemicals. Wash your hands well before leaving the laboratory. Also, wash your hands or any part of your body immediately with water when it comes in contact with chemicals.

4- Do not use your mouth to fill a pipette. There are special bulbs for this purpose.

5- Note the position of safety equipment like fire extinguishers, eye washers, and first aid boxes. Report all accidents immediately to a staff member or technician.

6- Use the fume hood when handling strong-smelling or irritating chemicals.

7- Be careful about discarding away wastes.Always follow instructions.

Do not dispose of solids into the sink. Do not leave glassware or any other solid materials, including filter papers, in the sink. Put broken glassware into the labeled buckets.

Some waste liquids must be stored into special bottles, not disposed of in the sink. A staff member or technician will help you.

8- Do not leave a lit burner unattended. Always stay clear form the flame.

9- When heating anything in a test tube, do not point the mouth of the test tube towards yourself or towards any other person.

10- Before leaving the laboratory turn off any water taps and burners and dispose of solid waste in the correct container. Also, wash all apparatus used and clean up the bench top.

11- Keep your bench clean and tidy while you are working. Clean up any spills or broken glass immediately. Keep your books and papers away from water, chemicals, and flames. Position your apparatus on the bench so that it is convenient and comfortable to use. Keep unused equipment out of the way, so that you do not knock it over.

12- If you are in any doubt about anything, ask the staff member or technician for advice.

13- Request the assistance of your instructor/technician if and when you suffer a cut or a burn **or face a dangerous** situation.
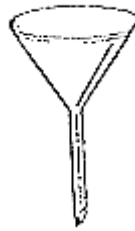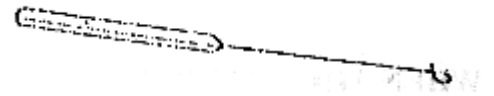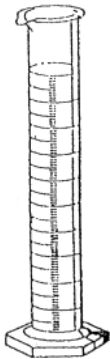
SOME LABORATORY APPARATUS
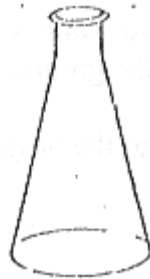
Beaker
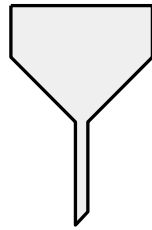
Büchner funnel

Funnel

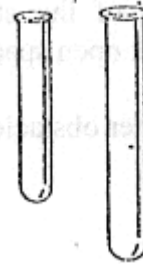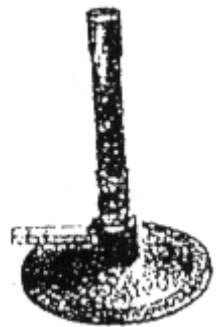Glass rod with platinum wire (for flame testing)

Graduated cylinder

Erlenmeyer flask

Dropping bottle

Test tubes

Bunsen burner
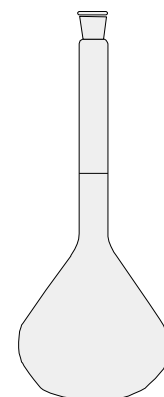
Stirring rod

Dropper

Safety glasses (Goggles)

Test tube rack
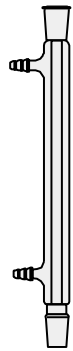
Plastic wash bottle

Clamp holder

Volumetric flask

Burette

Tripod	Condenser	Stand	Crucible	Volumetric pipette
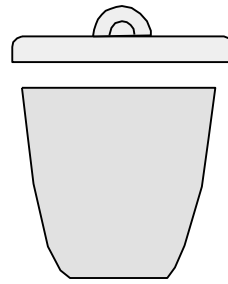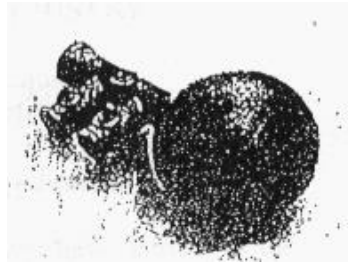
Reagent bottle	Pipette filler	Burette clamp
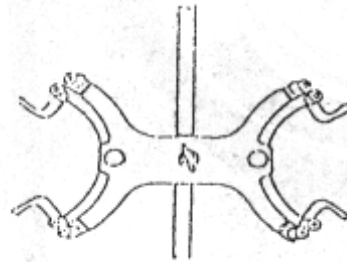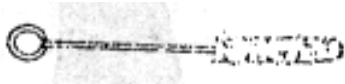
Test tube brush	Metal spatula	Test tube holder

Ring clamp	Ceramic square	Forceps
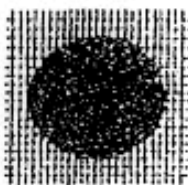
Wire gauze	Watch glass	Crucible tongs

Top-loading balance

FIRE REGULATIONS

Fire is a serious hazard in any laboratory. Always assume that an alarm is real. There is no need to leave the building, but only when you are instructed by your instructor to do so.

WHEN THE CONTINUOUS ALARMS SOUND:

1. Do not stay to collect personal belongings.

2. Follow the instructions of the staff and walk outside through the nearest exit to the nearest open space on the ground level.

3. If you meet smoke or other obstacles, go to the nearest clear exit.

WHEN THE INTERMITTENT ALARMS SOUND:

There is no need to leave the building, but be ready to leave if the alarm becomes continuous.

INSTRUCTIONS FOR ALL EXPERIMENTS

You may lose marks if you do not follow these instructions:

1. Wear a laboratory coat and safety glasses in the laboratory at all times.

2. Write all your results in the spaces provided in the laboratory manual at the time of the experiment.

3. Write all your results in blue or black ink, not in pencil.

4. If you make a mistake and you need to change an entry, don't erase it, overwrite it, or use correction fluid. Cross out the old value and write the new value as follows:

$$\text{Mass of water} = \quad \cancel{10.51} \quad 10.15 \text{ g}$$

5. Complete your experimental report, including calculations, in the laboratory. You should bring a calculator to the laboratory.

6. **Submit your report to your instructor for marking before you clean up your bench** and leave the laboratory.

7. When you have finished the experiment, clean your apparatus, and return everything that you took from your locker. **Leave your bench clean and tidy** when you go.

8. You may not work in the laboratory after the end of the laboratory period. Leave the laboratory on time so as to be able to reach your next class on time.

On this drawing, please mark the locations of the following equipment. There may be more than one item of each kind. Please mark them all. Walk around the lab to find each item. **You may need help from your instuctor** or a technician. Here is the list of items to find.

1. All exit doors	5. Fire blankets
2. Fume hoods	6. Emergency shower
3. Fire extinguishers	7. First aid kits
4. Eye wash stations	8. Poster with safety instructions
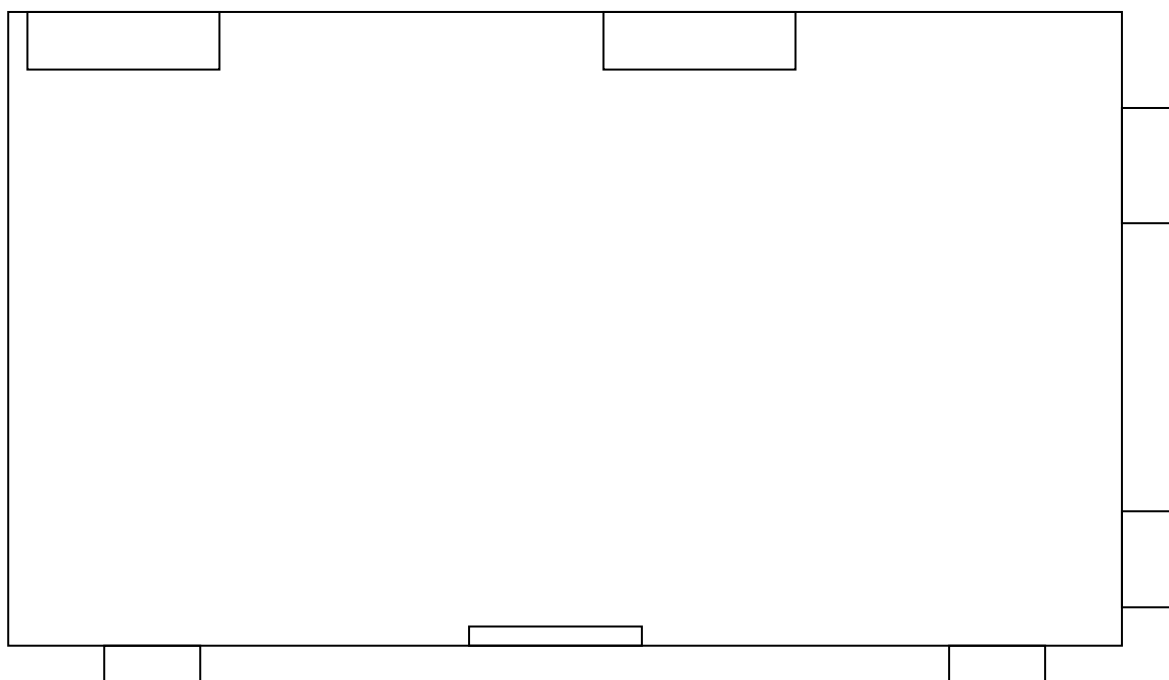
A. Safety

A chemistry lab could be a dangerous place to work in. To make sure that nobody gets hurt we ask you to follow a few simple instructions. You should already have read the safety instructions on pages 2,3 and 7. Please indicate that you have done so by signing your name below:

I have read and understood the 'Safety in the Laboratory' and the 'Fire Regulations' on   pages 2,3 and 7. I agree to follow these rules.

Name_____ID No._____

The lab has many safety features that can be used in an emergency. On the previous page you will find a drawing of the lab. Please mark the locations of all the safety equipment listed there.

B. Equipment

As in all new fields of study, there are some new words and equipment to get to know in the chemistry lab. Please take a few minutes to get familiar with the glassware and equipment you will be using during this course. Here is what to do:

Start by taking all items from your drawer and your locker and place them on the bench. Now take each item one by one and see if you can find its name on the list on pages 4, 5 and 6. If you find the item on the list, circle the drawing. Just ask if you need help.

Student Name: _____   Section No.: _____
  ID Number: _____   Date : _____
Partner Name: _____   Group No.: _____

Experiment (2)

Empirical Formula of a Compound

PreLab

1. How many grams of magnesium combine with 1.50 g of chloride ions in $MgCl_2$?

_____

_____

_____

_____

_____

2. If 11.80 g of iron reacts with 5.06 g of oxygen. Determine the empirical formula of the resulting oxide?

_____

_____

_____

_____

_____

_____

_____

_____

_____

Experiment (2)

Empirical Formula of a Compound

Objective:

- To determine the Chemical formula of magnesium oxide.

Introduction:

The empirical formula of a compound is the smallest whole – number ratio of atoms in the compound. The following are examples of empirical formulas: Fe_2O_3, ZnO, Mg_3N_2, CH_3, CH_2, CH, CO_2, MgS, and MnO_2. The empirical formula of a compound is usually determined either by chemical analysis (decompositions) or by synthesis. In the chemical analysis method, a known mass of the compound is decomposed (or burned) and the masses of the products are determined. These masses are converted to number of moles of the elements present in the compound. From the number of moles, the relative number of atoms of each element is determined, and this will give the empirical formula.

Example 1:

Nicotine is a compound containing C, H, and N. A 2.50 g sample of the compound is burned and produces 6.78 g of CO_2, 1.94 g of H_2O, and 0.43 g of N_2. what is the empirical formula of nicotine?

First we calculate the number of moles of each element present.

mole C = mole CO_2 = $.\dfrac{6.78g}{44.0g/mol}$ = 0.154 mole

mole H = 2 x mole H_2O = 2 x $.\dfrac{1.94g}{18.0g/mol}$ = 0.216 mole

mole N = 2 x mole N_2 = 2 x $.\dfrac{0.43g}{28.0g/mol}$ = 0.031 mole

ratio of moles = ratio of atoms

The empirical formula is $C_{0.154} : H_{0.216} : N_{0.031}$

Dividing by 0.031, (the smallest number) this will give the smallest whole-number ratio:

C_5H_7N = the empirical formula

In the chemical synthesis method, a known mass of a metal is combined with a non metal. The product is weighed and the mass of nonmetal is determined. From the mass of the metal and nonmetal, the number of moles is calculated and then the empirical formula is determined.

Example 2:

When 0.288 g of P is burned, 0.660 g of white phosphorus oxide is obtained, determine the empirical formula of this oxide?

mass of oxygen = 0.660 – 0.188 = 0.372 g

$$mole\ P = .\frac{0.288g}{31.0g/mol} = 0.0093\ mole$$

$$mole\ O = .\frac{0.372g}{16.0g/mol} = 0.0233\ mole$$

mole P: mole O = 0.0093: 0.0233 = 2 : 5

Empirical formula is P_2O_5

In today's experiment you will prepare magnesium oxide by burning a known mass of magnesium in air (O_2). From the mass of product, and magnesium, the empirical formula of magnesium oxide is determined.

Apparatus and Chemicals:

Balance, Bunsen burner, Crucible and cover, Clay triangle, Crucible tongs, Iron ring, clamps, stand, Mg ribbon and Steel wool.

Experimental Procedure:

Record all observations and data directly on report sheet IN **INK.**

1. Place a clean, dry, partially covered crucible on the clay triangle. Heat with hot flame for two or three minutes. This step removes any moisture from the crucible. See fig (1) for correct assembly.

2. Turn off the flame and allow the crucible and cover to cool on the clay triangle.

3. WHEN THE CRUCIBLE STOPS FEELING WARM TO YOUR HAND, bring the crucible and cover to a balance and weigh them to two decimal places($\pm$ 0.01 g). If the crucible is too hot you will not get an accurate weight.

4. Polish about 2 cm of Mg ribbon and cut it into small pieces. Use steel wool for polishing. Place about 0.25 g of the element in the crucible and weigh the crucible + Mg accurately to ($\pm$ 0.01 g) .

5. Replace cover and heat the crucible and contents slowly at the beginning. Lift the cover occasionally to introduce oxygen to react with Mg. Do not lift the cover widely, because too much air will burn the Mg brightly (with flame). If this happens cover the crucible immediately.

6. Continue heating until all Mg is converted to ash, then remove the cover and heat the crucible until the crucible bottom becomes red.

7. Remove the burner and Cool the crucible, add a few drops of distilled water to decompose any magnesium nitride, Mg_3N_2, that may have formed during combustion.

8. Heat the open crucible slowly until the ash is completely dry.

9. Allow the crucible to cool for 2 minutes on the clay triangle. Then place the covered crucible in the desiccators and cool to **room temperature**, about 15 to 20 minutes. **DO NOT PLACE THE HOT CRUCIBLE ON THE LAB BENCH AS IT WILL BURN THE BENCH**.

10. When the crucible cools to room temperature (that is, not warm to the touch), transfer it with the cover to the balance and weigh them to two decimal places.
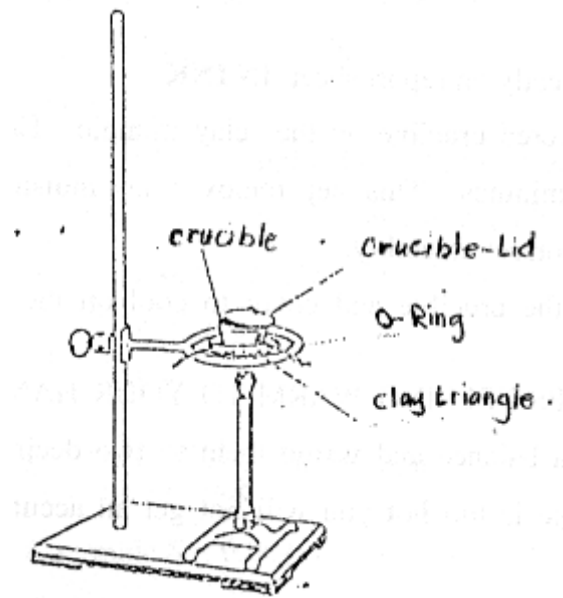
Fig. (1): Setup for magnesium oxide determination.

Student Name: _____   Section No.: _____

ID Number: _____   Date : _____

Partner Name: _____   Group No.: _____

Experiment (2)

Empirical Formula of a Compound

Report Sheet

Data:

		Trial I	Trial II
1.	Mass of empty crucible	_____	_____
2.	Mass of Mg	_____	_____
3.	Mass of Crucible and oxide	_____	_____
4.	Mass of magnesium oxide	_____	_____
5.	Mass of oxygen	_____	_____
6.	Moles of Mg (FM of Mg = 24.3 g /mole)	_____	_____
7.	Moles of oxygen (FM of O = 16.0 g/ mole)	_____	_____
8.	Mole Mg: Mole O	_____	_____
9.	Empirical formula of magnesium oxide	_____	_____
10.	% of O in the compound	_____	_____
11.	% of Mg in the compound	_____	_____

Show your calculation for steps of 6-11 :-

Experiment (2)

Empirical Formula of a Compound

PostLab

1. What is the effect of Mg_3N_2 if it is not decomposed on the reported (**Mg to oxygen**) mole ratio?

_____

2. Describe the effect of each of the following factors, whether increases, decreases, or has no effect on the reported value of (Mg to O) mole ratios:

   a. If carbon is deposited on the crucible's surface (because of improper heating) and the crucible with contents is weighed without removing the carbon residue by further heating.

   _____

   b. If the magnesium oxide ash is not dried completely.

   _____

   c. If rapid oxidation of magnesium occurs by too much air, and some Mg is lost.

   _____

   d. If air is not sufficient to react with all the magnesium.

   _____

3. Explain how magnesium nitride is formed during heating? Write a chemical equation for its formation.

   _____

4. Explain, by a chemical equation, the effect of the added water in the decomposition of magnesium nitride.

   _____

   _____

Experiment (3)

Limiting Reactant

Prelab

1. A 25.00 g sample of $Na_3PO_4.12H_2O$ reacts with excess $BaCl_2.H_2O$. If the mass of $Ba_3(PO_4)_2$ obtained is 17.56 g. Calculate the % yield of $Ba_3(PO_4)_2$?

$$2Na_3PO_4.12H_2O + 3BaCl_2.2H_2O \rightarrow Ba_3(PO_4)_{2(s)} + 6NaCl + 30H_2O$$

_____

_____

_____

_____

_____

_____

_____
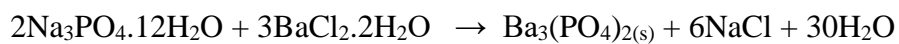
Experiment (3)

Limiting Reactant

Objectives:

- To determine the limiting reactant in a salt mixture.

- To determine the percent composition of a salt mixture.

Introduction:

In a chemical reaction, the reacting materials are often mixed in stoichiometric quantities (according to fixed mole ratios), i.e., in exactly the correct amounts so that all reactants are used up during the reaction.

When reactants are not mixed in the correct mole ratio, only limited amount of product is formed, and this amount is determined by the reactant present in less amount and thus is consumed first in a reaction. This reactant is called the limiting reactant.

Limiting reactant: It is the reactant that is consumed first, and thus determines the amount of product formed.

The theoretical yield of a product is the maximum amount that can be produced with a given amount of the limiting reactant. The ***actual yield*** is often less than the theoretical yield. The % yield is represented as:

$$\% \text{ yield} = \frac{\text{Actual yield}}{\text{Theoretical yield}} \times 100\%$$

Let us discuss the concept of limiting reactant by examining the reaction between $Na_3PO_4.12H_2O$ and $BaCl_2.2H_2O$.

This is a precipitation reaction which produces $Ba_3(PO_4)_{2(s)}$. The equation in molecular form is:

$$2Na_3PO_4.12H_2O + 3BaCl_2.2H_2O \rightarrow Ba_3(PO_4)_{2(s)} + 6NaCl + 30H_2O$$
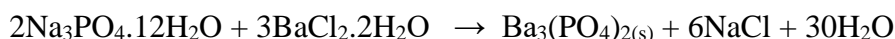
and the ionic equation is:

$$6Na^+ + 2PO_4^{3-} + 24H_2O + 3Ba^{2+} + 6Cl^- + 6H_2O \rightarrow Ba_3(PO_4)_{2(s)} + 6Na^+ + 6Cl^- + 30H_2O$$

The net ionic equation of the reaction is:

$$3Ba^{2+} + 2PO_4^{3-} \rightarrow Ba_3(PO_4)_{2(S)}$$
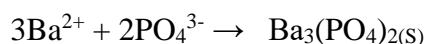
In this reaction:
3 moles of Ba^{2+} ions react with 2 moles of PO_4^{3-} ions, or
3 moles of $BaCl_2.2H_2O$ react with 2 moles of $Na_3PO_4.12H_2O$

Example 1:

A mixture containing 40.00 g of $Na_3PO_4.12H_2O$ and 30.00 g of $BaCl_2.2H_2O$ is dissolved in water. A precipitate of $Ba_3(PO_4)_2$ weighing 22.65 g is produced. Calculate the % yield of $Ba_3(PO_4)_{2(s)}$.
(FM) of $Na_3PO_4.12H_2O$ = 380.2 g/mole; (FM) of $BaCl_2.12H_2O$ = 244.2 g/mole.
The net ionic equation is:

$$3Ba^{2+} + 2PO_4^{3-} \rightarrow Ba_3(PO_4)_{2(s)}$$
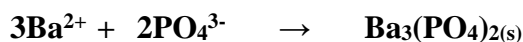
mole PO_4^{3-} = mole $Na_3PO_4.12H_2O$ = 40.00g /380.2 g mole^{-1} = 0.105 mole
mole Ba^{2+} = mole $BaCl_2.2H_2O$ = 30.00 g / 244.2 g mole^{-1} = 0.123 mole
0.105 mole PO_4^{3-} requires 0.105 x 3/2 = 0.185 mole Ba^{2+}. Since we have only 0.123 mole of Ba^{2+}, so the **limiting reactant** is Ba^{2+}, and the **reactant in excess** is PO_4^{3-}.

The **theoretical yield** of product is calculated from the limiting reactant as follows:
   mole $Ba_3(PO_4)_2$ = moles Ba^{2+} x $\underline{1 \text{ mole } Ba_3(PO)_2}$
                                            3 mole Ba^{2+}

$$= 0.123 \text{ x } \frac{1}{3}$$

$$= 0.041 \text{ mole}$$

mass of $Ba_3(PO_4)_2$ = 0.041 mole x 602.2 g mole^{-1} = 24.69 g (this is the theoretical yield)

% yield $= . \dfrac{22.65 g}{24.69 g}$ x 100 = 91.7 %

Example 2:

10.0 g of a unknown mixture containing $Na_3PO_4.12H_2O$ and $BaCl_2.2H_2O$ is dissolved in distilled water. The mass of $Ba_3(PO_4)_2$ precipitated is 3.50 g. Calculate the % of each salt present in the mixture if $BaCl_2$ is the limiting reactant?

$$3Ba^{2+} + 2PO_4^{3-} \rightarrow Ba_3(PO_4)_{2(s)}$$
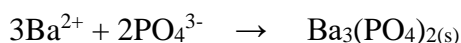
mole $Ba_3(PO_3)_2$ $= \dfrac{3.50 g}{602.2 g / mol}$ = 5.81 X 10^{-3} mole

mole Ba^{2+} = mole $BaCl_2.2H_2O$ = 3 x (mole $Ba_3(PO_4)_2$)

mole $BaCl_2.2H_2O$ = 5.81 X 10^{-3} x 3 = 1.74 x 10^{-2} mole

mass of $BaCl_2.2H_2O$ = 1.74 x 10^{-2} mole X 244.2 g mole^{-1} = 4.25 g

mass of $Na_3PO_4.12H_2O$ = 10.0-4.25 = 5.75 g

% $BaCl_2.2H_2O$ = $\dfrac{4.25}{10.0}$ x 100 = 42.5%

% $Na_3PO_4.12H_2O$ = $\dfrac{5.75}{10.0}$ x 100 = 57.5%

In the following experiment you will receive an unknown mixture of $BaCl_2.2H_2O$ and $Na_3PO_4.12H_2O$. The mixture is weighed and then dissolved in water, the precipitate formed is dried and weighed. From the weights of mixture and precipitate, the composition of the unknown mixture is determined.

Apparatus and Chemicals:

Balance	400- mL beaker
Bunsen Burner	100-mL beakers
Iron ring	Filter flask
Ring stand and clamps	Buchner funnel
Wire gauze	Filter paper (Whatman No. 42)
Watch glass	$BaCl_2$
Stirring rod	Na_3PO_4
Rubber policeman	Unknown mixture of ($BaCl_2.2H_2O$) and ($Na_3PO_4.12H_2O$)

Experimental Procedure:

Two trials are required. Weigh **duplicate** unknown salt mixtures and perform the following steps for each.

(1) Precipitation of Ba₃(PO₄)₂:

1. Weigh on a weighing paper about 1.0 g of an **unknown mixture** containing $BaCl_2.2H_2O$ and $Na_3PO_4.12H_2O$. Report the mass to ±0.01 g.
2. Transfer to a 400 mL beaker, add 200 mL of distilled water and **stir the mixture for one minute.**
3. Cover the beaker with a watch glass and **heat gently** with stirring for 30 minute. **Do not boil the solution**, keep the temperature at about 80-90°C
4. Allow the precipitate to settle, then decant two 50mL volume of the supernatant liquid into two 100 mL beakers, labeled beaker 1& 2 to be used in part (II).
5. Weigh a filter paper (± 0.01 g) and seal it into the Buchner funnel with a small amount of distilled water. While the solution is hot, **filter the precipitate** by vacuum as shown in Fig.1. Transfer any precipitate remaining in the beaker into the funnel with a policeman and hot water.
6. **Wash the precipitate** with **two portions of distilled water**, remove the filter paper and put it in the oven and dry the precipitate at a temperature of 100°C for about 1 hour.
7. Remove the filter paper from the oven and weight the precipitate and paper to (± 0.01 g)

(2)   Determination of the limiting reactant:

The limiting reactant is determined using the solutions in beakers 1 & 2 as follows:
Test for excess PO_4^{-3}: add 2 drops of 0.5 M $BaCl_2$ to the solution in beaker 1. If a precipitate is formed, then PO_4^{3-} is in excess and Ba^{2+} is the limiting reactant.
Test for excess Ba^{2+}: add 2 drops of 0.5 M Na_3PO_4 to the solution in beaker 2.
If a ppt. is formed, then Ba^{2+} is in excess and PO_4^{3-} is the limiting reactant.
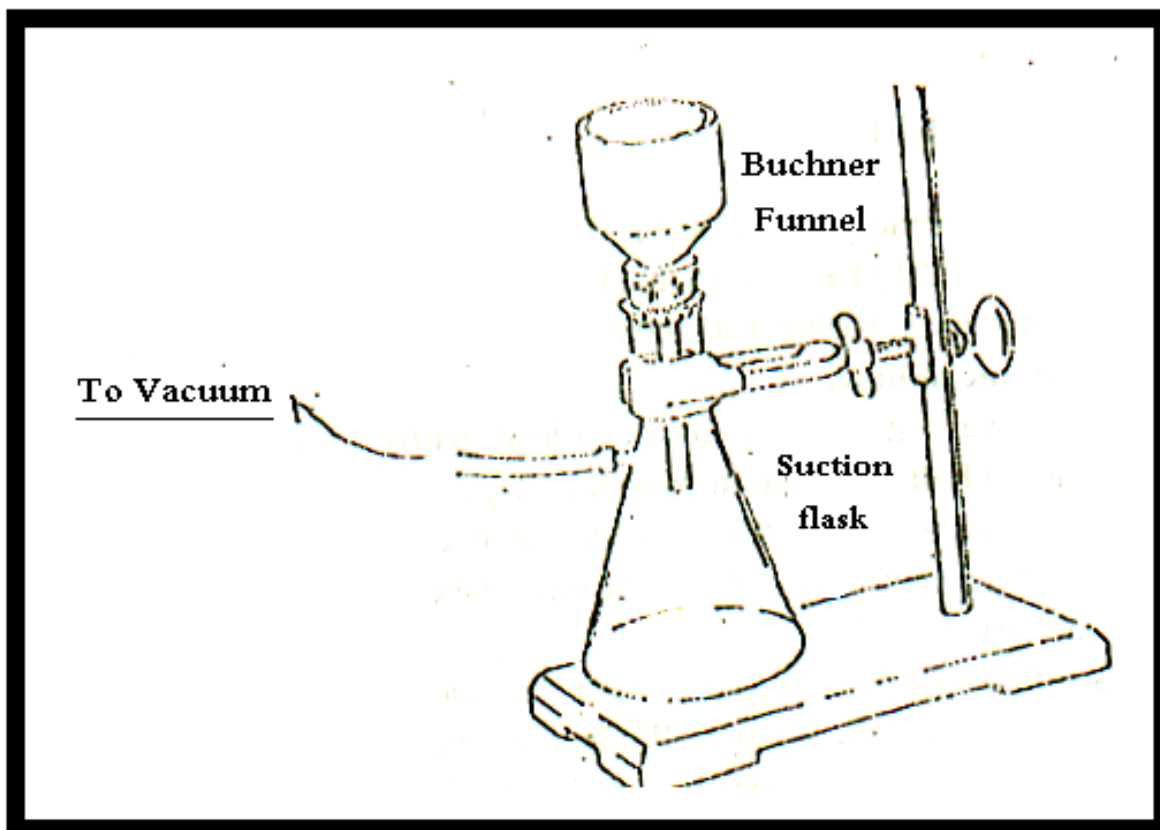
Fig. (1): Setup for vacuum filtration

22

Experiment (3)

Limiting Reactant

Report Sheet

Part (1):- Preparation **of $Ba_3(PO_4)_2$**

Unknown number   _____

Data:	Trial 1	Trial 2
1. Mass of mixture (g)	_____	_____
2. Mass of filter Paper (g)	_____	_____
3. Mass of Filter paper + $Ba_3(PO_4)_2$ (g)	_____	_____
4. Mass of $Ba_3(PO_4)_2$ (g)	_____	_____

Part (2): - Determination of the Limiting reactant

Data:	Trial 1	Trial 2
1. Limiting reactant in salt mixture	_____	_____
2. Excess reactant in salt mixture	_____	_____

Calculations:

	Trial 1	Trial 2
1. Mass of $Ba_3(PO_4)_2$ (g)	_____	_____
2. Moles $Ba_3(PO_4)_2$: (FM = 602.2 g/mole)	_____	_____
3. Moles $BaCl_2.2H_2O$; (FM = 244.2g/mole)	_____	_____
4. Mass of $BaCl_2.2H_2O$ reacted (g)	_____	_____
5. Moles $Na_3PO_4.12H_2O$ reacted; (FM = 380.2 g/mole)	_____	_____
6. Mass of $Na_3PO_4.12H_2O$ reacted (g)	_____	_____
7. Mass of salt mixture (g)	_____	_____
8. Mass of excess reactant (g)	_____	_____
9. % Of $BaCl_2.2H_2O$ in the mixture	_____	_____
10. % Of $Na_3PO_4.12H_2O$ in the mixture	_____	_____

Show your calculation on a separate paper.

Experiment (3)

Limiting Reactant

PostLab

1. What is the effect of heating the solution on the particle size of $Ba_3(PO_4)_2$ precipitate?

_____

_____

_____

2. Describe the effect of each of the following factors (whether increase, decrease, or has no effect) on the actual yield of $Ba_3(PO_4)_2$

   a. Using a coarse filter paper instead of one with fine porosity.

_____

_____

   b. Insufficient washing of the precipitate.

_____

   c. If the precipitate is washed with an acidic solution.

_____

   d. If the precipitate was not dried completely.

_____

Experiment (4)

Identification of a Compound: Physical Properties

Prelab

1. Define:-

   A. Solubility:

   _____

   _____

   _____

   B. Boiling point:

   _____

   _____

   _____

2. A students liquid unknown boils at approximately 69°C, is insoluble in water but soluble in cyclohexane. Its density is 0.65g / ml. Which chemical in table (1) is the unknown?

   _____

   _____

   _____

3. What physical property, measurable in this experiment, distinguishes *cyclohexane* from *cyclohexene*?

   _____

   _____

   _____

4. Using the apparatus described in this experiment, when should the boiling point of a liquid be recorded?

   _____

   _____

   _____

Experiment (4)

Identification of a Compound: Physical Properties

Objective:

- To identify a compound based on its physical properties
- To learn how to properly assemble a boiling point apparatus

Introduction:

Gold is yellow, roses are red, salt is white and crystalline, lead is "heavy" and water is a colorless clear liquid. These are all physical properties of chemical substances. Physical properties are often used for the identification of a chemical substance .The more common physical properties include color, odor, density ,solubility ,melting point, boiling point ,and whether the substance is a solid, liquid ,or gas. These properties can be observed without any knowledge of the reactivity of the compound. Additional tests, tests that reveal more information about its chemical or physical properties, may be necessary, however, to confirm its purity.

The purpose of this experiment to identify a chemical compound from the data collected on its solubility, density, and boiling point.

Solubility:

The solubility of a compound is the maximum mass (usually in grams) of the substance that dissolves in a fixed mass (usually 100g) of solvent at a given temperature. A chemical has different solubilities in different solvents, reflecting the differences in the molecular composition of the solute from that of the solvent. For instance, some substances such as table salt, are soluble in water but insoluble in gasoline. In this experiment we will examine the solubility of a substance in three different solvents: water, cyclohexane, (C_6H_{12}) and ethanol (C_2H_5OH). Qualitatively each substance is recorded as being soluble (complete dissolution), slightly soluble (partial dissolution), or insoluble.

Density:

The density is the mass per unit volume. Substances with a large density have a large mass in a small volume. While we commonly say that Lead is"heavy" we really mean that lead has a high density-it doesn't require much volume in order to have a large mass of lead.

Boiling point:

When a liquid is gradually heated, there is a temperature at which bubbles form spontaneously and continue to form until the entire volume of the liquid has been converted to a gas. This "constant" temperature is called the boiling point of the liquid. At the boiling point the temperature of the escaping vapor equals that of the liquid; this temperature depends upon the prevailing atmospheric pressure. The boiling point values listed in Table (1) are measured at normal atmospheric pressure (called one atmosphere of pressure).

The boiling point of a substance is characteristic of the magnitude of the forces acting between molecules, called ***intermolecular forces***. The greater the magnitude of the intermolecular forces, the higher will be boiling point. Since the magnitude of the intermolecular forces is different for each compound, each has a characteristic boiling point.

Apparatus and Chemicals:

75 mm test tube (4), dropper, 150 ml-beaker, pipette, thermometers 110ºC, rubber band, and capillary tube. Beaker 250 ml, Burner, wire gauze, O-ring. Stirring rod, Ethanol, Cyclohexane, Acetone, and about 15ml of an unknown liquid.

Experimental Procedure:

Note: (Ask your instructor for an unknown listed in Table 1)

A. Solubility:

1. **Solubility in Cyclohexane**:

   Into a 75-mm test tube add 3 drops of your liquid unknown to 1 ml of cyclohexane.Agitate the contents (Figure 1) Does each drop dissolve? Partially dissolve? Record your observations.

2. **Solubility in water and Ethanol**:
   Repeat the test with water and ethanol as solvents. Describe the solubility of your unknown as soluble, slightly soluble, or insoluble in each solvent.

Table (1) physical properties of some common laboratory chemicals
Symbols used: i = insoluble, sls = slightly soluble, s = soluble

Compound	Density(g/ml)	Boiling Point(C)	Solubility		
			H_2O	C_6H_{12}	C_2H_5OH
Acetone	0.79	56	s	s	s
2-butanone	0.805	80	s	s	s
Cyclohexane	0.79	80.74	i	-	s
Cyclohexene	0.81	83	i	s	s
Ethanol	0.79	79	sls	s	-
Ethylacetate	0.90	77	s	s	s
Heptane	0.684	98	i	s	s
n-hexane	0.66	68	i	s	s
1-hexene	0.67	63	i	s	s
Isopropanol	0.79	83	s	s	s
Methanol	0.79	65	s	s	s
n-propanol	0.805	97	s	s	s
Water	1.00	100	-	i	s

Tap–tap–tap
w/pinky
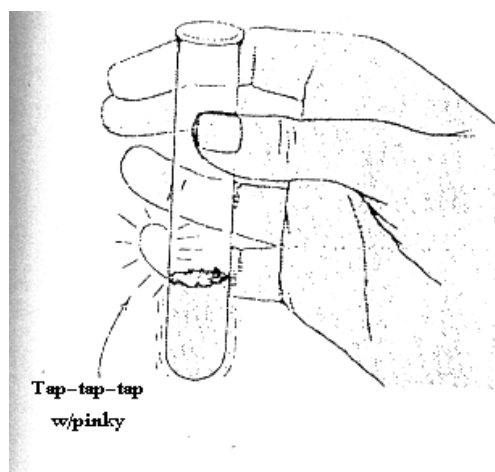
Figure (1) shake contents of the test tube

B. Density:

Complete two trials to determine the density of your unknown.
 Determine Mass and Volume of Unknown. Pipette 2ml of the liquid into a beaker and weigh. Calculate the density of the liquid.

C. Boiling point:

Check out a 110°C thermometer. Assemble the apparatus in Figure 2. Complete two measurements for the boiling point of your unknown.

1. Assemble the Boiling Point Apparatus. Place 10 drops of the unknown liquid into a 75-mm test tube. Position it beside the thermometer bulb using a rubber band. Invert (open end down) a 10-cm capillary tube in the liquid. Place the apparatus into the water bath.

2. Measure the Boiling Point. Slowly heat the water in the water bath while stirring with the stirring rod . When a rapid and continuous stream of bubbles escapes the capillary tube, discontinue heating the water bath. The stream of bubbles slows as the bath cools. When the bubbles cease to escape and before the liquid re-enters the capillary tube, record the temperature. This is the boiling point of the liquid.

3. Repeat the Measurement. Determine the boiling point a second time. The same (volume of) liquid may be used. However, the capillary tube must be removed and re-inserted before heating resumes.
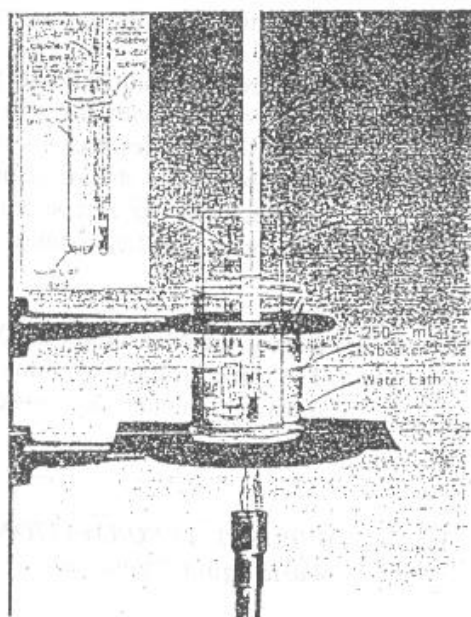
Figure (2) apparatus for determining the boiling point of a liquid

Student Name: _____   Section No.: _____

ID Number: _____   Date : _____

Partner Name: _____   Group No.: _____

Experiment (4)

Identification of a Compound: Physical Properties

Report Sheet

Solubility:

Unknown Number————

	Soluble	Slightly Soluble	Insoluble
1. Solubility in cyclohexane	_____	_____	_____
2. Solubility in ethanol	_____	_____	_____
3. Solubility in water	_____	_____	_____

Density:

	Trial 1	Trial 2
1. Volume of liquid (ml)	————	————
2. Mass of liquid (g)	————	————
3. Density of liquid (g/ml)	————	————
4. Average density of liquid (g/m)	————	

Boiling point:

1. Observed boiling point (c)	————	————

Name of unknown: _____

Experiment (4)

Identification of a Compound: Physical Properties

Post Lab

1. How does atmospheric pressure affect the boiling point of a liquid? Ask your instructor.

_____

_____

_____

2. If several drops of liquid unknown (Part B) cling to the pipette wall after delivery will the density of the unknown be reported too high or too low? Explain

_____

_____

_____

3. (**A**). In part C.2 if the boiling point is recorded when bubbles are rapidly escaping the capillary tube, will it be recorded too high or too low? Explain.

_____

_____

_____

   (**B**). If the boiling point is recorded after the liquid enters the capillary tube (after the heat is removed), will it be recorded too high or too low? Explain

_____

_____

_____

Experiment (5)

Tests for Cations and Anions

Objective:

- To observe reactions of several common cations and anions.
- To use chemical tests to identify an unknown salt.

Introduction:

One of the most common and important tasks for a chemist is to identify unknown compounds. In this experiment you will use simple chemical tests to identify an unknown salt containing one cation and one anion. We have limited the possible ions in your unknown to the following:

Cations	Anions
potassium, K^+	Sulfate, SO_4^{2-}
calcium, Ca^{2+}	Hydrogen carbonate, HCO_3^-
iron(III), Fe^{3+}	Chloride, Cl^-
ammonium, NH_4^+	Bromide, Br^-

Your unknown could be any of the 16 possible combinations of these cations and anions, for example KBr, $FeCl_3$, $Ca(HCO_3)_2$, $(NH_4)_2SO_4$, etc.

Chemicals and equipment:

0.1M solutions of:

sodium sulfate , Na_2SO_4 Two test tube holders
potassium chloride ,KCl   Bunsen burner
sodium chloride , NaCl   Platinum wire at end of glass rod
sodium bromide , NaBr   Red litmus paper
sodium hydrogen carbonate, $NaHCO_3$ Adhesive labels
silver nitrate , $AgNO_3$ Unknown salt
barium chloride , $BaCl_2$ Test tube rack with test tubes
sodium hydroxide , NaOH
sodium oxalate , $Na_2C_2O_4$
iron(III) sulfate , $Fe_2(SO_4)_3$
potassium thiocyanate , KSCN
calcium nitrate , $Ca(NO_3)_2$
ammonium nitrate , NH_4NO_3
2M nitric acid, HNO_3
2M hydrochloric acid, HCl

The procedure used in these tests is an example of **qualitative** analysis. You do not need to measure out volumes exactly using a measuring cylinder. In fact, doing so, would only waste time and increase the chance of contamination. Volumes of 1 or 2 mL are used. If you want to know how much this will fill a test tube, you can measure out 1 or 2 mL of distilled water in a measuring cylinder and pour it into a test tube. In the tests you can use approximately the same volumes of the solutions.

Use **clean** test tubes. Dirty glassware will give confusing or misleading results. After use, wash the test tubes several times with tap water and finally twice with distilled water from your wash bottle.

Each test is performed with a known compound and with your unknown salt in two test tubes held side by side so that the results can be compared. A positive test is a test that confirms the presence of a certain ion. When you perform the test on the known compound you will, of course, obtain a positive test. When you do the same test on your unknown you may see the same reaction, a different reaction or no reaction at all. Write down what you see using a complete sentence. For example: "A white precipitate formed", "The solution remains colorless", "A clear blue solution forms", etc. <u>Do not write</u> 'No change.' **You get a positive test for your unknown if you get the same result as was obtained for the known compound.** You should label each test tube to remember which tube contains the known compound and which contains the unknown.

To gain experience you should do all the tests for the cations and the anions even if you have already established the identity after a few tests. You can do the tests in any order.

Experimental Procedure:

A. Prepare a solution of your unknown

Place four spatula tips of your unknown sample in a 100 mL conical flask and add about 20 mL distilled water. Swirl to dissolve the salt. If you have difficulty in dissolving the salt, try to heat it in a hot water bath. Use this unknown solution in the following tests. More solution can be made up later if necessary.

B. Tests for cations

1. Test for calcium ion, Ca^{2+}

Take two clean test tubes. Place about 2 mL of calcium nitrate solution (known solution of calcium ion) in one test tube and about 2 mL of your unknown solution in the other test tube. Add about 10 drops of the sodium oxalate solution to each tube. Record your observation and conclusion below.

Solution	Observation	Conclusion
$Ca(NO_3)_2$		Ca^{2+} is present
Unknown		

2. Test for iron(III), Fe^{3+}

Take 2 mL of iron(III) sulfate in one test tube and 2 mL of your unknown solution in another test tube.
Add about 10 drops of potassium thiocyanate (KSCN) solution to each test tube. Shake to mix. Record your observations and conclusion below.

Solution	Observation	Conclusion
$Fe_2(SO_4)_3$		Fe^{3+} is present
Unknown		

3. Test for ammonium ion, NH_4^+

Place 2 mL ammonium nitrate solution in one test tube and 2 mL unknown solution in another test tube.

Add 1 mL sodium hydroxide solution to each tube.

Warm each tube in a hot water bath for about one minute. Hold a moistened (wet) piece of red litmus paper at the mouth of each test tube, taking care not to touch the side of the tube as you are testing for any gas evolved.

Solution	Observation	Conclusion
NH_4NO_3		
Unknown		

Conclusion from the cation tests

Unknown No. _____

The cation _____

C. Tests for anions

1. Test for sulfate ion, SO_4^{2-}

In one test tube place 2 mL sodium sulfate solution and in another tube 2 mL of your unknown solution.

Add 10 drops of hydrochloric acid to each tube, shake gently and then add about 10 drops of barium chloride solution. Record your observations and conclusion below.

Solution	Observation	Conclusion
Na_2SO_4		
Unknown		

2. Test for hydrogen carbonate ion, HCO_3^-

Place 2 mL sodium hydrogen carbonate solution in one test tube and 2 mL of your unknown in another test tube.

Add 2 mL hydrochloric acid to each tube and watch carefully for any **immediate** reaction. Record your observations and conclusion below.

Solution	Observation	Conclusion
$NaHCO_3$		
Unknown		

3. Test for chloride and bromide ions, Cl⁻ and Br⁻

Take **three** clean, labeled test tubes. Place 2 mL sodium chloride solution in one test tube, 2 mL sodium bromide solution in another test tube and 2 mL of your unknown in the third test tube.

Add about 10 drops of nitric acid and about 5 drops of silver nitrate solution to each tube. If you get a precipitate, look carefully at the color of the precipitate.

Solution	Observation	Conclusion
NaCl		
NaBr		
Unknown		

Conclusion from the anion tests

Unknown No. _____

The anion _____

Final conclusion

The formula of salt No. _____   is   _____

The name of the compound is: _____

Experiment (6)

Molar Mass of a Volatile Liquid

Prelab

1) For which of the following compounds can we determine its molar mass using the method described in this experiment? Give reasons. Benzene (b.p.78°C), Glycerol (b.p.180°C).

_____

_____

_____

_____

_____

2) A cylinder contains compressed hydrogen gas and the mass of the hydrogen is 20.0 g. What mass of oxygen would be contained in an identical cylinder at the same temperature and pressure?

_____

_____

_____

_____

_____

_____

_____

Experiment (6)

Molar Mass of a Volatile Liquid

Objective:

To determine the molar mass of an unknown volatile liquid.

Introduction:

According to Avogadro's principle, equal volumes of gases at the same temperature and pressure contain equal number of particles (molecules). Dumas used this principle in 1826 to determine atomic and molar masses. Dumas measured the densities of volatile liquids (easily vaporized), and from these densities he determined the atomic and molar masses of many gases. In this experiment the molar mass of a volatile liquid is determined using the modified Dumas apparatus and procedure. A volatile liquid is transferred to a weighed Erlenmeyer flask. The flask is then heated to the boiling point of water to vaporize the liquid and expel air and excess liquid. Finally the flask is cooled to room temperature. The flask with contents is weighed again and the mass of the condensed vapor is calculated. The molar mass is determined using the ideal gas law:

$$PV = nRT \qquad\qquad (1)$$

The number of moles, n, is given by

$$n = (m/M) \qquad\qquad (2)$$

The ideal gas equation becomes:

$$PV = (m/M)\ RT \qquad\qquad (3)$$

Where: P = atmospheric pressure (atm)

V = volume of vapor (L)

m = mass of vapor (g)

M = molar mass of volatile liquid (g/mol)

R = gas constant = 0.0821 atm L K^{-1} mol^{-1}

T = temperature of boiling water (K).

Experimental Procedure:

1. Weigh together a clean and **dry** 125 ml Erlenmeyer flask, a copper wire and an aluminum foil (8 x 8 cm) on a Top-loading balance and record the weight as accurately as possible.

2. Using a graduated cylinder, pour about 10 ml sample of the volatile liquid (supplied by your instructor) into the Erlenmeyer flask.

3. Center the aluminum foil (8 x 8 cm) over the mouth of the flask and secure it in place with the copper wire.

4. Place a larger piece of aluminum foil (10 x 10 cm) over the first foil, cover and secure it in place with a rubber band.

5. Make a small hole through the aluminum foils with a sharp pin.

6. Place the Erlenmeyer flask into a 400 ml beaker and clamp it to a stand.

7. Place some water in the beaker, to cover up the liquid level in the flask, along with few boiling stones.

8. Place the beaker, with the flask over Bunsen burner as shown in the following figure.

9. Heat the beaker <u>slowly</u> till the water boils (this step should take about 10 minutes).

10. Allow the water to boil <u>gently</u>, till all the volatile liquid has vaporized. Continue gentle heating of water for another 5 minutes.

11. Measure the temperature of the boiling water and record it.

12. Turn off the Bunsen burner.

13. Using a towel, immediately remove the **Erlenmeyer** flask from the beaker.

14. Dry the outside of the flask and the aluminum foil **completely.**

15. Remove the <u>outer</u> aluminum foil **and rubber band** <u>only</u>.

16. Allow the Erlenmeyer flask to sit for 15 minutes to cool to room temperature.

17. While waiting for the Erlenmeyer flask to cool, read the atmospheric pressure on the laboratory barometer.

18. Weigh the Erlenmeyer flask, with aluminum cover and wire still on, accurately and record the mass.

19. Determine the volume of the flask by filling it to the rim with water and measuring the volume of the water with a graduated cylinder. Record the volume.

20. Repeat the above steps for a second trial.
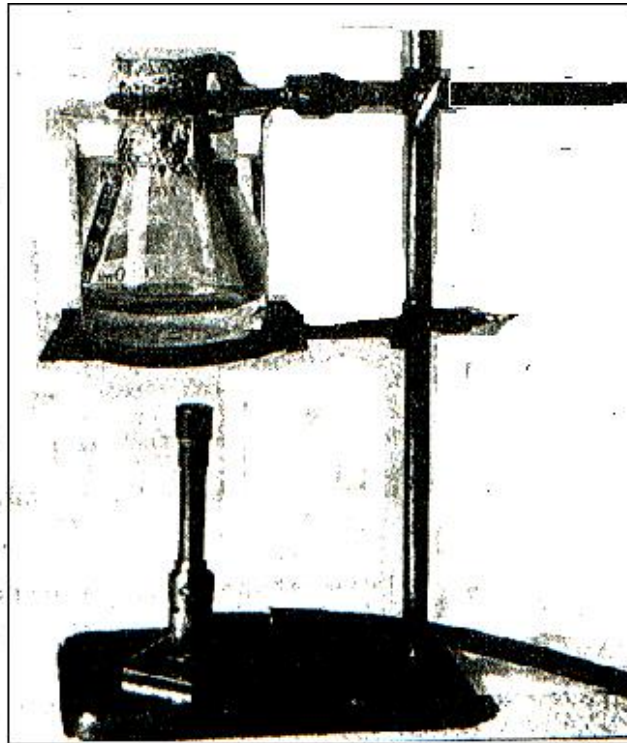
.

Experiment (6)

Molar Mass of a Volatile Liquid

Report Sheet

Unknown No. _____

Data:

		Trial I	**Trial II**
1.	Mass of empty Erlenmeyer flask + Aluminum foil + Wire (g)	_____	_____
2.	Temperature of boiling water (K)	_____	_____
3.	Atmospheric pressure (atm)	_____	_____
4.	Mass of Erlenmeyer flask + Condensed vapor + aluminum foil + wire (g)	_____	_____
5.	Mass of condensed vapor (g)	_____	_____
6.	Volume of Erlenmeyer flask	_____	_____
7.	Molar mass of the liquid, calculated	_____	_____
8.	Average molar mass	_____	

Note: *A more accurate calculation of the molar mass uses a corrected mass for the vapor which takes into consideration the mass of air displaced by vapor at room temperature.*

Experiment (6)

Molar Mass of a Volatile Liquid

PostLab

1. A flask weighs 40.1305g when clean, dry, evacuated, 138.2410g when filled with water (density = 0.9970 g/ml) and 40.2487g when filled with a gaseous substance at 470.4 torr and 96ºC. What is the molar mass (g/mol) of the gas? [R = 0.082 L.atm/mol. K]

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

Student Name: _____   Section No.: _____

ID Number: _____   Date : _____

Partner Name: _____   Group No.: _____

Experiment (7)

<u>Determination of the Molar Volume of Hydrogen Gas</u>

PreLab

A student at The Hashemite University wants to determine experimentally the volume occupied by one mole of H_2 gas at STP. She reacts 0.1471 g of Zn with excess HCl*(aq)* and collects 56.09 ml of gas over water at 22 °C and 757.8 torr. The vapor pressure of water at 22 °C is 19.8 torr.

1. Use the data given above to calculate:
 (i) The volume occupied by one mole of dry H_2 at 22 °C and 760 torr.
 (ii) The volume occupied by one mole of dry H_2 at STP.
Note: do NOT simply use the ideal gas law equation!

2. Name the gas laws which you used in your calculations.

Experiment (7)

Determination of the Molar Volume of Hydrogen Gas

Objectives:

- To determine the volume, in liters, of one mole of dry hydrogen (H_2) gas at STP

Introduction:

Hydrogen gas, or dihydrogen, (H_2) can be produced from the reaction of active metals with acids. In this experiment you will generate H_2 by reacting magnesium metal with hydrochloric acid. You will collect the H_2 in an inverted burette by water displacement and measure the volume of the gas. You can then calculate the volume of dry gas at STP if you know the room temperature, the barometric pressure, and the vapor pressure of water. We know that the ideal gas law predicts a volume of 22.4 L mol^{-1}, but in this experiment we want to *measure* this for ourselves.

Apparatus and Chemicals:

50 mL burette, one- or two-hole stopper to fit burette, ring stand with clamp, 600 mL beaker, 10 mL and 1000 mL graduated cylinders, fine copper wire, 6 M HCl, 5-6 cm piece of magnesium ribbon.

Experimental Procedure:

1. The first step is to record the volume contained in your burette between the 50 mL mark and the sealed end. It is written on your burette

2. Weigh a piece of magnesium ribbon with a mass of approximately 0.035–0.04 g on an analytical balance. Record its mass to four decimal places.

3. Fold the magnesium ribbon into a small pellet. Wrap it tightly all around with copper wire so that no small pieces can escape as it dissolves. Leave about 5 cm of copper wire to serve as a handle. See Figure 1.

4. Clamp your burette in an upright position in the ring stand. Fill a 600 mL beaker about two-thirds full of tap water and place near the ring stand.

5. Measure about 10 mL of 6 M HCl in a graduated cylinder. Tilt the burette at an angle from the upright position and pour in the HCl.

6. With the burette in the same position, SLOWLY fill it with tap water from the beaker. While pouring, rinse any acid that may be on the sides of the tube so that the liquid in the top of the tube will contain very little acid. TRY TO AVOID STIRRING UP THE ACID LAYER IN THE BOTTOM OF THE TUBE.

7. While holding the copper coil by the handle, insert the metal about 3 cm down into the burette. Hook the copper wire over the edge of the tube and hold it there by inserting the stopper. Fill the burette completely so that the stopper displaces a little water when put in place. See the left of Figure 1.

8. Cover the hole in the stopper with your finger and invert the burette in the 600 mL beaker of water, as shown in the middle of Figure 1. Clamp the burette in place. The acid being more dense than water will flow down to the bottom and eventually react with the metal.

9. After the reaction stops, wait for about 5 minutes to allow the burette to come to room temperature. If any bubbles stick to the sides of the burette, tap them gently.

10. Cover the hole in the stopper with your finger and transfer the burette to a large graduated cylinder almost full of room temperature water. See Figure 2. Raise or lower the burette until the level of the liquid inside the burette is the same as the level outside the burette. This permits you to measure the volume of the gases in the burette (H_2 and water vapor) at room pressure.

11. Read the burette. The calibrations are upside down, so make sure you read it correctly. Ask for help if you are not sure of the reading. Record this reading on your data sheet.

12. Remove the burette from the water and pour the acid solution it contains down the sink. Rinse the burette with tap water and then with distilled water.

13. Record the temperature of the water in the large graduated cylinder, the room temperature, and the room pressure as given by your instructor.
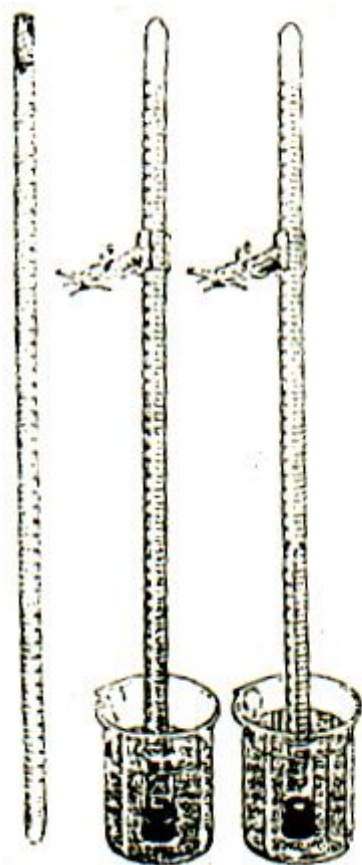
14. Do the experiment a second time.

Figure 1 Manipulating the burette.   **Figure 2 Measuring the volume of gas.**

45

Student Name: _____   Section No.: _____

ID Number: _____   Date : _____

Partner Name: _____   Group No.: _____

Experiment (7)

Determination of the Molar Volume of Hydrogen Gas

Report Sheet

	Trial 1	Trial 2
Volume of burette from 50 mL mark to the sealed end		
Mass of piece of magnesium ribbon		
Burette reading from Step 11		
Temperature of the water in the graduated cylinder (Step 13)		
Room temperature		
Room pressure		
Vapor pressure of water—use the water temperature from Step 13 and the Table below.		

Vapor Pressure of Water at Various Temperatures			
Temperature ℃	Pressure torr	Temperature ℃	Pressure torr
16	13.6	21	18.6
17	14.5	22	19.8
18	15.5	23	21.0
19	16.5	24	22.4
20	17.5	25	23.8

Calculations:

Set up each problem completely and **SHOW UNITS.** Cancel the units when you do the calculation. Use the correct number of significant figures in your answers.

1. Write the balanced equation for the reaction.

_____

2. Determine the number of moles of magnesium used. Find the average for the 2 trials.

_____

_____

3. Determine the volume of gas produced. Find this by the following equation:
   Volume of gas = (Volume of burette from 50 mL to sealed end) + (50.0 mL – burette reading). Find the average for the 2 trials.
   Use the average of your data from the previous page for these calculations.

_____

_____

4. Determine the partial pressure of the H_2 gas. Since the H_2 is collected over water, the gas in the burette consists of a mixture of H_2 and water vapor. The total pressure is $P_{room} = P_{H_2} + P_{H_2O}$.

_____

_____

_____

5. Using the results of your calculations in the previous steps, determine the volume (in liters) of the H_2 at one atmosphere pressure (760 torr) if no water were present.

_____

_____

_____

6. Using the results of your calculations in the previous steps, calculate the molar volume (in liters/mol) of dry H_2 at room temperature and 1 atmosphere pressure. (*Don't* simply use the ideal gas law, use your own results!)

_____

_____

7. Using the results of your calculations in the previous step, calculate the molar volume (in liters/mol) of dry H_2 at STP.

Student Name: _____

ID Number: _____   Date : _____

Partner Name: _____   Group No.: _____

Experiment (8)

Colligative Properties: Molar Mass Determination

PreLab

1. Students prepared two cyclohexane solutions having the same mass of solute. However Student 1 used 13 g of cyclohexane, Student 2 used 15 g. Which student will observe the larger freezing point change? Explain.

_____

_____

_____

2. A 0.597 g sample of a non-electrolyte dissolves in 20.0g of cyclohexane. The freezing point depression is 3.62 °C. What is the molar mass of the non-electrolyte? (K_f for Cyclohexane is 20.0 °C Kg/mol).

_____

_____

_____

_____

_____

_____

_____

Experiment (8)

Colligative Properties: Molar Mass Determination

Objective:
 To determine the molar mass of a non-volatile, non-electrolyte by observing the difference between the freezing points of a solvent and a solution.

Introduction:
 The addition of a non-volatile solute to a solvent produces characteristic changes in a solvent's physical properties. These changes are: **vapor pressure lowering**, **boiling point elevation** and **freezing point depression**. For example when antifreeze (ethylene glycol) is added to an automobile's cooling system it will prevent 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.

 Vapor pressure lowering, boiling point elevation, and freezing point depression are called **colligative properties** of solutions. These properties are governed by the number, rather than the type, of solute particles dissolved in the solvent.

 The change in colligative properties of a solution is directly proportional to the amount of solute dissolved in the solvent. The freezing point depression, ΔT_f, and the boiling point elevation, ΔT_b, are proportional to the molality, m, of the solute in the solution:

$$\Delta T_f \quad = \quad K_f\, m \tag{1}$$

$$\Delta T_b \quad = \quad K_b\, m \tag{2}$$

Where

 K_f and K_b are the molar freezing point and boiling point constants for the solvent.

$$m = \frac{mol\ \ solute}{Kg\ solvent} = \frac{(g\,/\,mol.wt.)solute}{Kg\ solvent} \tag{3}$$

 In this experiment you will determine the molar mass of a non-volatile, non-electrolyte (solute) in cyclohexane by measuring the freezing points for the pure cyclohexane and the solution, as well as the mass of solute and solvent. From K_f and equations (1) and (3), you will calculate the formula weight of the solute.

 Cyclohexane's freezing point and that for the solutions are obtained from a cooling curve; a graph representing a decreasing temperature as a function of time (Figure 1). Cyclohexane's cooling curve reaches a plateau at its freezing point; extrapolation of the plateau to the temperature axis determines its freezing point. The solution's cooling curve does not reach a plateau, but continues to decrease slowly as the cyclohexane freezes. Its freezing point is determined at the intersection of two lines drawn tangent to the curves above and below the freezing point (See Figure 1).

Experimental Procedure:

A. Freezing Point of Solvent:
1. Assemble the apparatus shown in Figure (2). Weigh (±0.01 g) a clean, dry 200 mm test tube. Add approximately 10 g of cyclohexane to the test tube and reweigh (±0.01 g) the tube and contents. Prepare about 300 mL of an ice-water slurry in a 400-mL beaker. Place the test tube and cyclohexane in the ice-water bath (Figure 2). Insert a thermometer, through one hole of a two-hole stopper, into the solvent to measure the temperature.
2. While stirring with the wire stirrer inserted through the stopper's other hole, record temperature (± 0.1°C) readings at timed intervals (30 sec) on the Report Sheet. The temperature remains virtually constant at the freezing point until solidification is almost complete. Continue recording until the temperature begins to drop again. On a linear graph paper (provided by your laboratory technician), plot the temperature (°C, vertical axis) versus time (t, horizontal axis) to obtain cyclohexane's "cooling curve". The temperature corresponding to the plateau of the cooling curve is cyclohexane's freezing point.

B. Freezing Point of Solvent-Unknown Solute:

Two freezing point trials for the solution are to be completed. The same unknown solute-cyclohexane solution can be used to repeat Parts B.1 and B.2.
1. Dry the outside of the test tube containing the cyclohexane and reweigh (±0.01 g). Ask the instructor for a solid unknown sample. Record your unknown number on your data sheet. Weigh (± 0.01 g) approximately 0.2g of the unknown on weighing boat. Transfer the unknown to the cyclohexane in the 200-mm test tube. In the transfer be certain that **none** of the solid solute remains in the boat or adheres to the test tube's side. If some does, roll the test tube until the solute dissolves. If an unknown is a liquid, weigh (±0.01 g) about 3 mL of it in a 10-mL graduated cylinder, pour this into the cyclohexane; then reweigh the empty cylinder. The difference is the weight of the unknown liquid added to the cyclohexane.
2. Determine the solution's freezing point in the same way as the solvent's was found in Part A. When the solution is near the freezing point, record the temperature (±0.1°C) at more frequent intervals (20 sec). Plot the temperature vs. time (on the same graph as in Part A) to get the solution's cooling curve. It will show a "break" at the temperature where freezing began. The break may not be as sharp as that for the pure solvent. Draw lines tangent to the curves above and below the freezing point; the lines' intersection is the solution's freezing point. Repeat the freezing point determination one more time (warm the tube and the solution to a temperature higher than the freezing point of the solution then cool again as before.) Have your instructor approve your graphs. After completing the collection of the experimental data, discard the solution into a bottle marked "waste solvent."
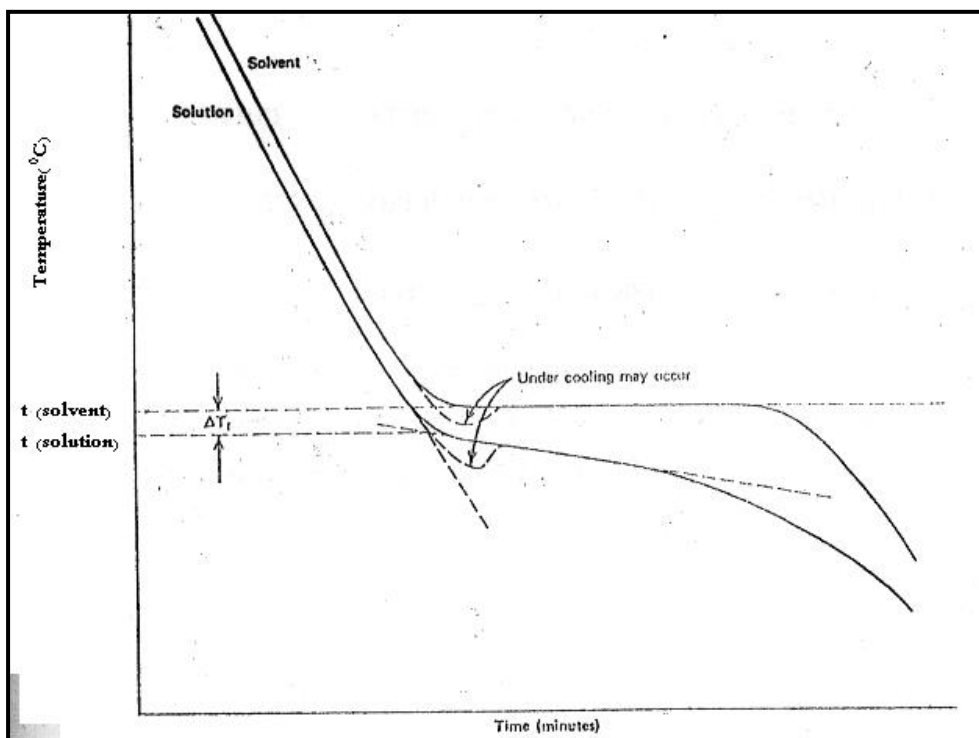
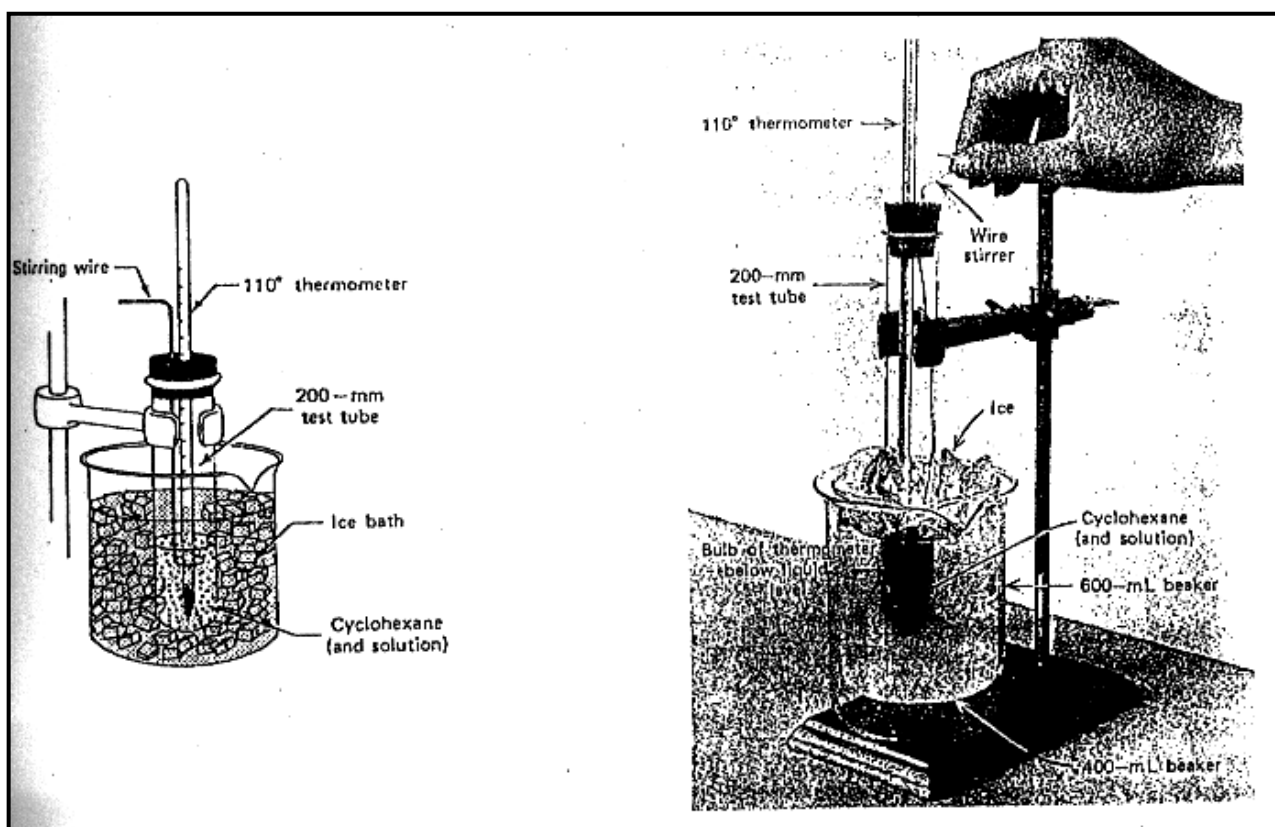**FIGURE 1. Cooling curves for solvent and solution**

FIGURE 2. Freezing point apparatus

Student Name: _____   Section No.: _____

ID Number: _____   Date : _____

Partner Name: _____   Group No.: _____

Experiment (8)

Colligative Properties: Molar Mass Determination

Report Sheet

Unknown Number _____

Data:

A. Freezing Point of Cyclohexane

1. Mass of test tube (g)   _____

2. Mass of cyclohexane (g)   _____

3. Freezing point, from cooling curve (°C)   _____

B. Freezing Point of a Solvent-Unknown Solute	Trial I	Trial II
1. Mass of test tube + cyclohexane (g)	_____	_____
2. Mass of cyclohexane (g)	_____	_____
3. Mass of solute (g)	_____	_____
4. Freezing point, from cooling curve (°C)	_____	_____

Calculations:

	Trial I	Trial II
1. Freezing point constant, Kf, of cyclohexane	20.0° C.kg/mol	
2. Freezing point change, ΔT_f (C°)	_____	_____
3. Mass of solute in solution (g)	_____	_____
4. Mass of cyclohexane in solution (kg)	_____	_____
5. Molar mass (g/mol)	_____	_____
6. Average molar mass (g/mol)	_____	

DATA FOR COOLING CURVES					
PART A (SOLVENT)		PART B (SOLUTION)			
Trial 1		Trial 1		Trial 2	
Time(second	Temp.	Time	Temp.	Time	Temp.
0		0		0	
30		20		20	
60		40		40	
90		60		60	
120		80		80	
150		100		100	
180		120		120	
210		140		140	
240		160		160	
270		180		180	
300		200		200	
330		220		220	
360		240		240	
390		260		260	
420		280		280	
450		300		300	
480		320		320	
510		340		340	
540		360		360	
570		380		380	
600		400		400	
630		420		420	
660		440		440	

➢ If needed, continue recording your data on separate sheets of papers and submit them with your Lab Report.

Experiment (8)

Colligative Properties: Molar Mass Determination

PostLab

1. If the solution's freezing point is erroneously read 0.2°C lower than it should be, will the unknown's calculated molar mass be too high or too low ? Explain!

    _____

    _____

2. How will the freezing point change of cyclohexane be affected (compared with the freezing point change by a non-volatile, non-associating and non-dissociating solute) by:

    (a) A non-volatile solute that dissociates? Explain.

    _____

    _____

    (b) Two solutes that react according to the equation,

    $$A + B \rightarrow C?$$ Explain!

    _____

    _____
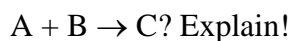
3. If some solute adheres to the test tube's wall in Part B.1, is the freezing point change greater or less than it should be? Explain!

    _____

    _____

    _____

Experiment (9)

C a l o r i m e t r y

Objective :

To measure heat of reaction

Introduction :

This experiment is designed to measure the HEATS OF REACTION of a series of chemical reactions. There are several knids of "heats of reactions", such as heat of solution, heat of neutralization, heat of combustion, heat of formation, etc. However, we shall study only the first two of these.

1. *Heat of solution*: it is the heat flow during the process of solution; as an example when NaOH dissolves in water, hydrated ions are formed and heat is released. The solution absorbs this heat and, therefore increases in temperature.

$$NaOH_{(s)} \xrightarrow{H2O} H_2O \qquad Na^+_{(aq)} + OH^-_{(aq)} + Heat \qquad \Delta H_1$$
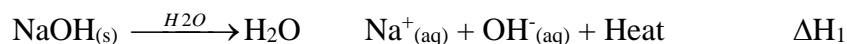
Where the heat of solution in kJ is ΔH_1

2. *Combined heat of solution and heat of neutralization*: in a second reaction, solid sodium hydroxide is reacted with an aqueous solution of hydrogen chloride. During this reaction, water and a dilute aqueous solution of sodium ions and chloride ions are formed. This time both the heat of solution and the heat of neutralization are released:

$$NaOH_{(s)} + H^+_{(aq)} + Cl^-_{(aq)} \quad \rightarrow \quad H_2O + Na^+_{(aq)} + Cl^-_{(aq)} + Heat \qquad \Delta H_2$$
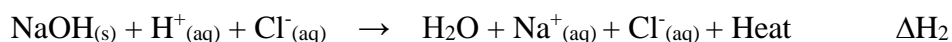
Where the combined heat of solution and heat of neutralization in kJ is ΔH_2.

3. *Heat of neutralization*: It is the heat evolved, at constant pressure, when acids are neutralized with bases. **It** can be obtained from the two steps above. The difference between the **heat of reactoin** obtained by dissolving solid NaOH in aquesous HCl solution (step 2) and the **heat of solution** obtained by dissolving solid NaOH in water (step1).

4. The final experiment of this laboratory class will be to determine the heat of solution of an unknown salt, as in (step 1) above.

Example 1

A 2.00 g sample of solid CsOH is dissolved in 200.0 mL of water in a calorimeter. The temperature of the water was raised from 22.3 to 23.4 °C. Calculate the heat of solution in kJ/mol. (assume the specific heat of the solution to be 4.184 J/g °C and the density of the solution to be 1.00 g/mL).

    a. Temperature change, ΔT= 23.4- 22.3 = 1.1 °C

    b. Total mass of solution, m, (200.0 mL water) (1.00 g/mL) + 2.00 g CsOH =202.0 g

    c. Heat absorbed by the solution = heat evolved by CsOH =

       Mass of solutin x specific heat of solution x ΔT =

       202.0 g x 4.184 J/g °C x 1.1°C = 930 J

    d. Moles of CsOH = mass/Molar mass = 2.00 g / 149.9 g /mol = 0.01334 mol

    e. Heat evolved per mole of CsOH = 930 J/0.01334 mol = -7.0 x 10^4 J/mol

    f. Heat of solution in KJ/mole = (-7.0 x 10^4 J/mol) x (1 kJ/10^3 J) = -70 kJ/mol

Note: the minus sign shows that the reaction is exothermic; i.e heat is given off (evolved) by the system.

Example 2

A 2.00 g sample of solid CsOH reacted with 200.0 mL of an aqueous solution of hydrogen chloride (HCl) in a calorimeter, the temperature of the solution increased from 22.3 to 24.3 °C. Calculate the heat of reaction in kJ/mol? (Assume the specific heat of the solution to be 4.184 J/g °C and the density of the solution to be 1.00 g/mL)

    a. Temperature change, ΔT = 2.0 °C

    b. Total mass of solution = 202.0g

    c. Heat absorbed by the solution = 202.0 X 4.184 X 2.0

$$= 1.7 \text{ X } 10^3 \text{ J}$$

    d. Moles of CsOH = 0.01334 mol

    e. Heat evolved per mole of CsOH = 1.3 x 10^5 J/mol

    f. Heat of reaction in kJ /mol = 1.3 X 10^2 kJ /mol

Note: The difference in the heat of reaction (Example 2) and the heat of solution (Example1) is the **heat of neutralization**. So the heat of neutralization of CsOH with HCl is 60 KJ /mol

Experimental Procedure:

Assume that the **density** of all solutions is 1.00 g/mL and their **specific heat** is 4.184 J/g ºC .

A.   Determination of the heat of solution for NaOH (ΔH_1)

1.   Add 100.0 mL of water to the calorimeter.

2.   Close the calorimeter and stir the water until a constant temperature is reached.

     Record the temperature of water.

3.   Weigh accurately about 2.0 g of NaOH. You should weigh the NaOH fast, since it is very HYGROSCOPIC.

4.   Add the solid NaOH to the water in the calorimeter. Stir gently. Read the temperature every 30 seconds. Record the maximum temperature reached.

B.   Determination of the heat of reaction (ΔH_2)

1.   Add 100.0 mL of 0.500 M HCl solution to the calorimeter.

2.   Stir the solution as in procedure 1 above, and record the initial temperature.

3.   Weigh accurately about 2.0 g of NaOH. You should weigh the NaOH fast, since it is very HYGROSCOPIC.

4.   Add the solid NaOH to the HCl solution in the calorimeter and stir gently until the maximum temperature is reached. Record the maximum (final) temperature.

C. Determination of the heat of solution of an unknown salt:

1.   Pick up an unknown salt from the instructor and record the unknown number on the report sheet. You will be given the molar mass of the salt.

2.   Repeat the procedure in part A, using 3.0 g of the unknown salt in place of NaOH.

Experiment (9)

C a l o r i m e t r y

R e p o r t   S h e e t

Part A: Determination of the heat of solution of NaOH (ΔH₁)

Data:

		Trial I	Trial II
1.	Volume of water	_____	_____
2.	Mass of water	_____	_____
3.	Mass of NaOH	_____	_____
4.	Molar mass of NaOH	40.0g/mol	
5.	Moles of NaOH	_____	_____
6.	Mass of solution	_____	_____
7.	Initial temperature	_____	_____
8.	Final temperature	_____	_____
9.	ΔH_1 of solution	_____	_____
10.	Average ΔH_1	_____	_____

Part B: Determination of the heat of reaction of NaOH with HCl (ΔH_2) and the heat of neutralization (ΔH_n)

    Data:

		Trial I	Trial II
1.	Volume of HCl solution	——————	——————
2.	Mass of HCl solution	——————	——————
3.	Mass of NaOH	——————	——————
4.	Molar mass of NaOH	40.0 g/mol	
5.	Moles of NaOH reacted	——————	——————
6.	Mass of solution	——————	——————
7.	Initial temperature	——————	——————
8.	Final temperature	——————	——————
9.	ΔH_2 of solution	——————	——————
10.	Heat of neutralization ($\Delta H_2 - \Delta H_1$)	——————	——————
11.	Average heat of neutralization	——————	

Part C: Determination of the heat of solution of an unknown salt:

    Data:

		Trial I	Trial II
1.	Volume of water	——————	——————
2.	Mass of water	——————	——————
3.	Mass of unknown salt	——————	——————
4.	Initial temperature	——————	——————
5.	Final temperature	——————	——————
6.	ΔH of solution	——————	——————
7.	Average ΔH	——————	

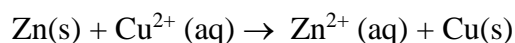**Show your calculation for Trial I:**

Experiment (10)

Electochemistry

Objectives:

- To measure the cell potential for various electrochemical cells.
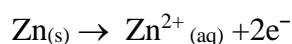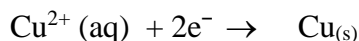- To study the dependence of the cell potential concentration on the cell.

Introduction:

A. Electrochemical Cells:

An oxidation-reduction reaction (redox reaction) such as:

$$Zn(s) + Cu^{2+} (aq) \rightarrow Zn^{2+} (aq) + Cu(s)$$

can be separated into two half-reactions, called half-cell reactions.

$$Cu^{2+} (aq) + 2e^- \rightarrow Cu_{(s)}$$
$$Zn_{(s)} \rightarrow Zn^{2+}_{(aq)} + 2e^-$$

The first half-reaction is the reduction half-cell and the second half-reaction is the oxidation half-cell.
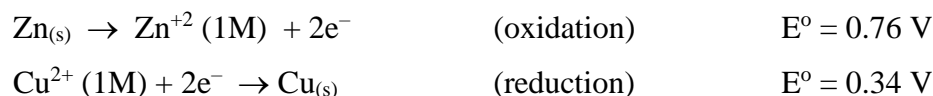
The above redox reaction can be carried out in a way such that the components of the oxidation and reduction half-cells are not mixed together, and the potential of the reaction can be measured, as shown in the Figure below.

Such a cell can be represented by:

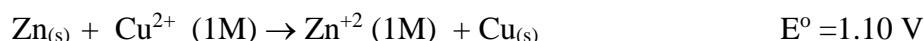$$Zn_{(s)} \mid Zn^{2+} (1.0M) \mid\mid Cu^{2+} (1.0M) \mid Cu(s)$$

The measured potential for this cell is 1.10 V. This value is referred to as the standard cell potential, E° which indicates the voltage observed when all species are in their standard states (1.0 M for species in solution, and 1.0 atm. pressure for gases). Most commonly E^{O} values are measured at 298 K.

This cell potential, E^o, can be calculated from the two half-cell potentials:

$$Zn_{(s)} \rightarrow Zn^{+2} \ (1M) \ + 2e^- \qquad \text{(oxidation)} \qquad E^o = 0.76 \ V$$

$$Cu^{2+} \ (1M) + 2e^- \rightarrow Cu_{(s)} \qquad \text{(reduction)} \qquad E^o = 0.34 \ V$$

Net reaction:

$$Zn_{(s)} \ + \ Cu^{2+} \ (1M) \rightarrow Zn^{+2} \ (1M) \ + Cu_{(s)} \qquad E^o = 1.10 \ V$$

When a cell reaction is written so that E^O is positive, the reaction will proceed spontaneously from left to right when all components are in their standard states.
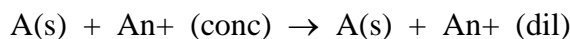
B. **Concentration Cells**

In this experiment you will observe the effect of varying the concentration of a species, participating in an electrochemical oxidation-reduction reaction, on the cell potential. Since the potential of an electrode depends on the concentration of the ions used in the electrode half-cell, a cell may be constructed from two half-cells differing only in the concentration of ions. The principle behind this is simple, dilution of a concentrated solution is spontaneous, so this process gives a cell with positive E. For the process:

An+ (aq) (concentrated) $\rightarrow$ An+ (aq) (dilute)

a cell can be constructed such as:

A | An+ (dil) || An+ (conc) | A

which represents the reaction:

A(s) + An+ (conc) $\rightarrow$ A(s) + An+ (dil)

Since the two half-reactions are the same, Eo(anode) = Eo(cath) so that Eo (cell) = 0.

Applying Nernst equation, at 25°C, we get:

$$E = -\frac{0.0592}{n} \log \frac{[A^{n+}]dil}{[A^{n+}]conc}$$

where n is the number of electrons transferred.

Experimental Procedure:

A. **Electrochemical Cells:**

1. Make a complete cell as shown in the Figure. Place 30 mL of 0.10 M $Zn(NO_3)_2$ solution and 30 mL of 0.10 M $Cu(NO_3)_2$ solution in separate 100 mL (or 150 mL) beakers.

   Obtain a zinc plate and a copper plate from the store room, clean both sides of each plate (electrode) with a strip of sand paper.

2. Fill a U tube with saturated KNO_3 solution, insert loose cotton plugs soaked with a saturated KNO_3 solution in the ends of the tube leaving *no air bubbles*. Place the U tube across the beakers as a salt bridge.

3. Insert the metal plates in the solutions of their own ions.

4. Attach the appropriate electrodes to the voltmeter's terminals, using connecting wires, and read the voltage.

5. Remove the wires connecting the cell to the voltmeter and short-circuit the cell with a single piece of wire.

   What chemical reaction is taking place?

   Which way are the electrons flowing?

   What changes can you observe which confirm that a chemical reaction is taking place?

6. Repeat steps 1-5 above making the following cells: Pb/Cu, and Pb/Zn. Record the voltage reading for each cell. (The standard reduction potential of $Pb^{+2} + 2e- \rightarrow Pb$ is -0.13 V)

B. **Concentration Cells**

Set up a simple concentration cell by using a Cu/0.10 M $Cu(NO_3)_2$ half-cell as that prepared in section A, and a second half-cell prepared as follows:

1. Take 30 mL (the same volume as before) of 0.1M KNO_3 solution and add to it exactly one drop of 0.10M $Cu(NO_3)_2$ solution.

2. Clean a Cu strip with a sand paper and insert it into the beaker.

3. Connect the two half-cells by a salt bridge and measure the cell voltage.

4. Now add 10 drops of 0.10M Na_2S solution to the second, more dilute, half-cell. Since CuS is very insoluble the addition of Na_2S has the effect of decreasing Cu^{2+} concentration to an extremely small value.

5. Measure the cell voltage again.

6. Label the electrode signs and processes (draw a diagram).

7. Measure the temperature of the solution.

Experiment (10)

Electochemistry

Report Sheet

Data and Calculations

A. **Electrochemical Cells:**

For each cell you made calculate Eo from your textbook table of electrode potentials. Compare your calculated values with those observed. Explain differences if any.

Cell	**Measured**	**Calculated**
Zn/Cu	_____	_____
Pb/Cu	_____	_____
Zn/Pb	_____	_____

Zn/Cu: Chemical reaction: _____

Direction of e- flow: _____

Pb/Cu: Chemical reaction: _____

Direction of e- flow: _____

Zn/Pb: Chemical reaction: _____

Direction of e- flow: _____

B. Concentration Cells

Calculations:

1. One drop has a volume of about 0.05 mL, thus the concentration of Cu2+ in the dilute half-cell is about (0.05/30) x 0.1M = 2 x 10-4M. Use the Nernst equation to calculate the value of E at 298 K and compare it with your observed value. Why might they differ?

$E_{observed}$ = _____

$E_{calculated}$ (show calculations) =

_____

_____

_____

_____

_____

2. After adding 10 drops (0.5 cm3) of Na2S solution (the concentration of S2- is about (0.5/30) (0.1M) = 2 x 10-3M), calculate the concentration of Cu2+ from the relationship of the solubility product:

$$Ksp = [Cu^{2+}] [S^{2-}] = 8 \times 10^{-37}.$$

Then calculate the value of E and compare it with the observed value obtained after the addition of Na_2S solution to the dilute Cu^{2+} half-cell.

$E_{observed}$ = _____

$E_{Calculated}$ (show calculations) =

_____

_____

_____

Experiment (11)

Chemical Thermodynamics

Objectives:

- To study the factors that affect the spontaneity of a chemical reaction.

- To determine some thermodynamic functions for a chemical reaction.

Introduction:

Several criteria could be used to judge the spontaneity of a chemical change. All these take into account both the enthalpy change of a reaction, Δ H, and the change in the entropy or randomness, Δ S, of the reaction. One simple measurement is the equilibrium constant, K.

Consider the reaction:

$$aA + bB \Leftrightarrow cC + dD$$
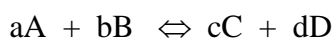
$$K = \frac{[Ce]^c \quad [De]^d}{[Ae]^a \quad [Be]^b}$$

Where [Ae], [Be], [Ce], [De] are equilibrium concentrations.

At any given concentration, the concentration expression, Q, is:

$$Q = \frac{[C]^c \quad [D]^d}{[A]^a \quad [B]^b}$$

K = Q only at equilibrium. By comparing K and Q values one can predict the direction of the spontaneous reaction. Usually, measurements of K are not easy to carry out.

A second quantitative criterion of the tendency for reaction to occur is the potential or electromotive force, EMF, of a oxidation-reduction reaction. A positive potential indicates that the reaction will proceed spontaneously in the direction as written and a negative potential indicates that the reaction will proceed spontaneously in the opposite direction.

A third criterion of the tendency of a reaction to occur is the free energy change of the reaction, ΔG^*.

Since potentials, equilibrium constants and free energies measure the tendency of a reaction to occur, they must be interrelated, and they are, through the expression:

$$\Delta G^\circ = -nFE^\circ = -RT \ln K$$

Where ΔG° is the standard free energy change of the reaction and E° is the standard cell potential.

If we know E^o for example, both K and ΔG^o can be calculated.

Cell potential measurements can often be made quickly and accurately and therefore are favored for finding the tendency of a reaction to occur, especially in aqueous solutions.

In this experiment you will measure EMF and ΔH for a chemical reaction. From these you will calculate ΔG and ΔS values.

* If ΔG is negative, the reaction will proceed spontaneously from left to right as written. If ΔG is positive, the reaction will proceed spontaneously in the opposite direction.

Experimental Procedure:

1. Prepare 25 mL of a solution containing 0.10M $Fe(NO_3)_3$ and 0.10M $FeSO_4$, by mixing 8.50 mL of 0.30M $FeSO_4$, 8.50 mL of 0.30 M $Fe(NO_3)_3$ and 8.00 mL of H_2O (using a graduated cylinder).

2. Introduce the 25 mL of this solution into a 150 mL beaker.

3. In another beaker, add about 0.5g of manganese dioxide, MnO_2, and introduce 25 mL of a ready made mixture of 0.10 M $KMnO_4$ and 0.1M HNO_3.

4. Prepare the salt bridge as you did in the previous experiment.

5. Insert the carbon electrodes in the beakers and measure the voltage of the cell consisting of the two half cells:

      i. $Fe^{2+} \rightarrow Fe^{3+} + e^-$

and

      ii. $MnO_4^- + 4H^+ + 3 e^- \rightarrow MnO_2 + 2H_2O$
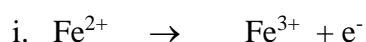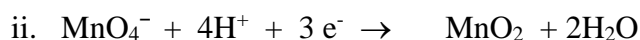
6. When the voltage is stabilized record it.

7. Calculate ΔG for the cell reaction using the relationship:

8. $\Delta G \text{ (kJ)} = -96.5 \, nE$

9. where E is the observed voltage in volts and n is the number of electrons exchange in the overall redox reaction..

10. Measure ΔH for the reaction as follows:

    a) In a 100 mL beaker, place 20mL of 0.20M $KMnO_4$, 5.0mL of 6M HNO_3 and 5.0mL of water, (all measured by graduated cylinder).

    b) Place 20mL of 0.50M $FeSO_4$ in a flask.

    c) Carefully measure the temperature of each solution ($KMnO_4$ and $FeSO_4$ solutions), to the nearest 0.2^oC.

    d) Add the $FeSO_4$ solution to the $KMnO_4$ solution in the beaker with stirring and record the highest observed temperature.

Experiment (11)

Chemical Thermodynamics

Report Sheet

Data and Calculations:

1. Voltage of the cell (V)   _____

2. Δ G for the cell reaction (kJ)   _____

3. Average temperature of solutions before mixing   _____

4. Maximum temperature after mixing   _____

5. Change in temperature (ΔT)   _____

6. Total volume of final solution   _____

7. Mass of solution (g) (assume density = 1.0 g/cm^3)   _____

8. Specific heat of solution   4.18J/ g°C

9. Heat absorbed by solution (kJ)   _____

10. Heat absorbed by the beaker (kJ)   _____

    N.B. Heat capacity of the beaker   (100 mL beaker)   30 J/°C

                                            (150 mL beaker)   40 J/°C

11. Heat evolved by the reaction (kJ)   _____

12. Calculate ΔH per mole of MnO_4^- reacted (calculate the initial moles of MnO_4^- and Fe^{2+}

    and note carefully which reagent limits the extent of the reaction).

    _____

    _____

    _____

    _____

    _____

13.   From the ΔG and ΔH values obtained, calculate ΔS for reaction under the conditions

       you used.

    _____

    _____

    _____

Experiment (12)

pH, Hydrolysis

PreLab

1. Which ion hydrolyzes more, Fe^{3+} , or Fe^{2+} ?_____. Explain.

_____

_____

_____

2. Write an equation representing the hydrolysis of each ion:

a. F^-: _____

b. Fe^{3+} : _____

c. HSO_4^- : _____

3. Predict whether an aqueous solution of each of the following salts is acidic, basic, or neutral:

Salt	Acidic, Basic, or Neutral	Equation justifying your prediction
Na_2CO_3		
KNO_3		
$AlCl_3$		
NH_4NO_3		

Experiment (12)

pH, Hydrolysis

Objectives:

- To measure the pH of laboratory and home acids and bases.

- To measure the extent of hydrolysis of various ions.

Introduction:

pH:

The acidity or basicity of most aqueous solutions is frequently due to small concentration of H^+ or OH^-. The concentration of H^+ or OH^- is expressed in terms of the pH, such that:

$$pH = — \log [H^+] \tag{1}$$

Water dissociates very slightly producing equal concentrations of H^+ and OH^- :

$$H_2O \iff H^+ + OH^- \tag{2}$$

At 25°C, the $[H^+] = [OH^-] = 1.0 \times 10^{-7}$ mol/L producing a pH = 7. Addition of an acid, which either releases H^+ ions or consumes OH^- ions, increases $[H^+]$ and thus pH becomes less than 7. Addition of a base, which either releases OH^- ions or consumes H^+ ions, decreases the $[H^+]$ in solution, producing a pH greater than 7.

In this experiment, the pH's of some acids and bases will be measured using acid-base indicators or a pH-meter.

Hydrolysis:

Salts ionize in aqueous solution forming cations and anions, which are surrounded by polar water molecules (hydrated) to varying degrees depending on the size and the charge of the ion. A strongly hydrated cation attracts the *negative* end of the polar H_2O molecule and weakens the O-H bond. This liberates free H^+, producing an acidic solution. The pH is then less than 7. A strongly hydrated anion, on the other hand, attracts the *positive* end of the H_2O molecule, again weakening the O-H bond and setting OH^- free, producing a basic solution. The pH is then greater than 7. This splitting of water molecules by ions is called *hydrolysis*.

Experimental Procedure:

A -   pH measurement with indicators:

1. Clean nine test tubes with soap and water and rinse with distilled water.

2. Place 2 mL of each solution listed in the Report Sheet into separate test tubes and add 5 drops of universal indicator to each. Estimate the pH of each solution.

   **pH measurement with pH-meter:**

1. Calibrate the pH meter. (This step is done by your laboratory technician).

2. In a 50-mL beaker introduce about 30 mL of each test solution listed in the Report Sheet.

3. Stir the sample, insert the electrodes, and record the pH. Allow sufficient time for the reading to reach a stable value.

4. Remove the electrode and the sample. Place a "waste" beaker under the electrodes, rinse them with distilled water, touch dry with a soft paper towel, and repeat steps 2 and 3 with the other samples described in Part A. Save the test samples for other students to use.

B -   Hydrolysis of Salts:

1. Transfer 30 mL of each solution listed in the Report Sheet into a 50-mL beaker.

2. Measure the pH of each solution with the pH-meter. Identify the ion that hydrolyzes and write an equation for its reaction with water.

C -   Atmospheric Hydrolysis:

Ammonium salts readily hydrolyze with water vapor releasing ammonia gas. The extent of hydrolysis may be detected by smelling the solid crystals. Cautiously smell solid NH_4Cl, $NH_4C_2H_3O_2$, and $(NH_4)_2CO_3$. The salt producing the strongest ammonia odor causes the equilibrium

$$NH_4^+ {}_{(aq)} + H_2O \rightarrow NH_{3(g)} + H_3O^+{}_{(aq)} \tag{6}$$
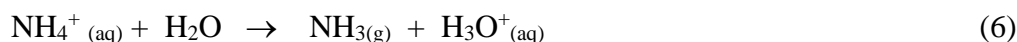
to shift farthest to the right. Therefore, the anion having the strongest affinity for hydronium ion (the strongest base) causes equilibrium (6) to shift farthest to the right, releasing more ammonia gas. Which anion, Cl^- , $C_2H_3O_2^-$, or CO_3^{2-} , is the strongest base?

Experiment (12)

pH, Hydrolysis

Report Sheet

A. pH

Solution	pH	
	Indicator	**pH meter**
0.10 M HCl		
0.10 M $HC_2H_3O_2$		
0.10 M NH_3		
0.10 M NaOH		
Distilled Water		
Tap Water		
Orange Juice		
Milk		
7-UP		

B. Hydrolysis of Salts

Solution	pH	Ion Hydrolyzed	Balanced Equation
	PH-meter		
0.1 M KCl			
0.1 M $NaC_2H_3O_2$			
0.1 M NH_4Cl			

C.   Atmospheric Hydrolysis

1.   List the ammonium salts in order of decreasing strength of ammonia smell.

_____

2.   List the anions in order of decreasing base strength.

_____

3.   Write an equation for each anion hydrolysis.

a. _____

b. _____

c. _____

Experiment (13)

Acid-Base Titration

Prelab

1-Define the following terms:-

a) Neutralization:

_____

_____

_____

b) Indicator:

_____

_____

_____

c) End Point:

_____

_____

_____

d) Equivalence point:

_____

_____

_____

2-In a titration of NaOH against HCl, 26.12 mL of 0.1624 M NaOH was used to neutralize a certain volume 0.1438 M HCl. What volume of HCl was neutralized?

_____

_____

_____
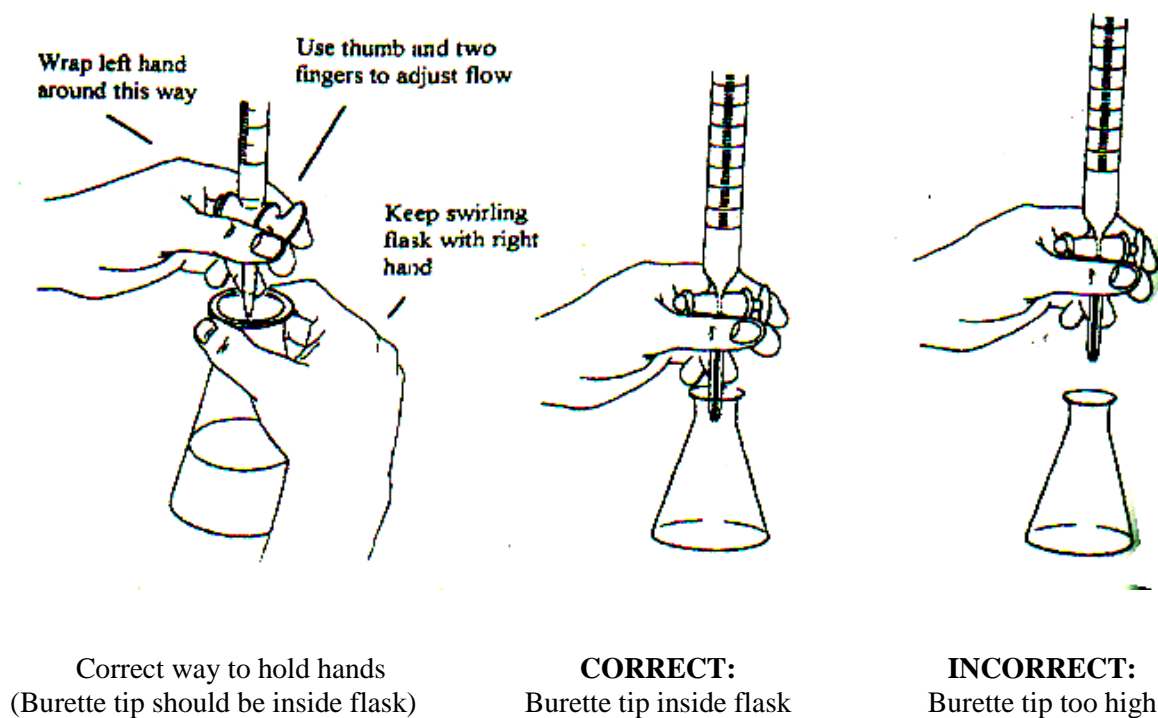
_____

_____

Experiment (13)

Acid-Base Titration

Objectives:

- To determine the standard concentration of sodium hydroxide solution using this standard solution of potassium hydrogen phthalate.
- To determine the concentration of an unknown hydrochloric acid (HCl) solution, using a standard solution of sodium hydroxide.
- To make accurate volume measurements using a pipette ,burette and volumetric flask.

Notes for good laboratory practice:

1. Always label beakers that contain your solutions.

2. Always rinse out the burette with soap & tap water, distilled water then three times with a small amount of titrant (in this experiment, sodium hydroxide).

3. Do **not** try to get the meniscus to a particular position, e.g. 0.00 mL. A piece of white paper with a black bar placed behind the glass may help you to read the volume.

4. After filling the burette, always remove the funnel. The funnel should **NOT** stay permanently at the top of the burette since any drops of liquid from the funnel will change your readings.

5. Check to see that no bubbles are in the tip of the burette.

6. When titrating, the tip of the burette must be inside the flask as shown below.

7. After draining titrant out of the burette, wait about 10 s and then read the burette.

8. Keep your eye level with the meniscus for all readings.

9. All titrations must be carried out a minimum of three times. The first titration gives an approximate value.

Your hands should hold the stopcock and flask as shown below

Wrap left hand around this way

Use thumb and two fingers to adjust flow

Keep swirling flask with right hand

Correct way to hold hands
(Burette tip should be inside flask)

CORRECT:
Burette tip inside flask

INCORRECT:
Burette tip too high

Experimental Procedure:

A. Making the KHP solution:

1. Weigh out, to 2 decimal places, about 0.50 g KHP. Use the spatula to transfer the crystals from the bottle to the 100 mL conical flask. Record the mass in the "Report sheet".

2. Add amount of distilled water to the KHP. Swirl carefully until all the solid has dissolved.

B. (1) Standardization of sodium hydroxide solution by titration:

1. Rinse out the burette with a small amount of sodium hydroxide solution. Throw the rinse solution away in the sink.

2. Fill the burette with the NaOH solution, using a small, funnel. Run out enough liquid so that the meniscus is below the zero mark. Make sure that no air bubbles are trapped in the tip of the burette. Remove the funnel from the burette.

3. Record the volume to **two decimal places** as the "Initial burette reading"
4. Add a few drops (about 2 drops) of the phenolphthalein indicator to the KHP solution.

5. Rough titration: Add the NaOH solution from the burette rapidly to the conical flask containing the KHP and indicator. Swirl the flask gently as you add the NaOH so that the solutions mix. When the pink color takes a longer time to disappear, add the NaOH more slowly. **Stop as soon as you see the first   permanent light pink color**

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6. Record the volume in the burette as the "Final burette reading". Calculate the "Volume added" by subtracting the initial burette reading from the final burette reading. This is the **approximate** volume of NaOH you need for the titration.

7. Rinse out the flask after each titration with distilled water. **Never rinse the flask with KHP solution.** First titration: Refill(if necessary) the burette with NaOH solution and make a note of the new "Initial burette reading" .Prepare your sample as in Steps 1&2 in part A. Take the volume from your rough titration and subtract 2 mL. You can add this volume of sodium hydroxide quickly to your flask. Then add the NaOH slowly and carefully (drop by drop) until the first permanent light pink color is seen. Record the "final burette reading" and calculate the "Volume added".

8. Repeat the titration for a second trial.

B. (2) Titration of an unknown (HCl) solution

Note: Ask your instructor for your unknown (HCl) solution

Repeat the steps in part B-1 using **unknown (HCl) solution** instead of the **KHP solution.**

Repeat the titration two more times

Finally, **clean all used glassware (including the burette) first with tap water, then with distilled water.**

Experiment (13)

Acid-Base Titration

Report Sheet

Data:

Part (B.1): Standardization of a sodium hydroxide by titration:

	Rough titration	First titration	Second titration
Mass of KHP			
Initial burette reading:			
Final burette reading:			
Volume added:			

 Average volume of NaOH solution added = _____ mL

Calculations:

1.   Write a balanced equation for this reaction. **NOTE: The KHC$_8$H$_4$O$_4$ has one acidic proton.**

_____

2. Calculate number of moles of KHP.

_____

_____

_____

3. Calculate the number of moles of NaOH.

_____

4. Calculate the concentration of the NaOH solution in mole L^{-1} in the average volume of the solution used in the titration.

_____

_____

_____

_____

Conclusion:
The concentration of NaOH solution is _____mole L^{-1}. (***To be used in part B.2***)

Part (B.2) :Titration of an unknown (HCl) solution:

	Rough titration	First titration	Second titration
Final burette reading:			
Initial burette reading:			
Volume added:			

Average volume of sodium hydroxide added = _____ mL

Number of the unknown hydrochloric acid solution: _____

Calculations:

1. Write a balanced equation for this reaction.

_____

2. Calculate the number of moles of sodium hydroxide in the average volume.

_____

_____

_____

_____

3. Calculate the number of moles of HCl in 10.00 mL of the hydrochloric acid solution.

_____

_____

4. Calculate the concentration of the HCl solution.

_____

_____

_____

_____

Conclusion:

The concentration of Unknown #_____ HCl solution is _____ mole L^{-1}.

Experiment (13)

Acid-Base Titration

PostLab

1. What is the effect of the following (increase, decrease or no effect) on the determination of the concentration of HCl?

a) Air bubbles in the burette.

_____

_____

b) More than the required drops of Phenol naphthalene is added to the flask.

_____

_____

c) The color at the end point is too pink.

_____

_____