



CHEM 114

GENERAL CHEMISTRY

LABORATORY-II

BOOKLET

2022-2023 SPRING SEMESTER

PREFACE

Üniversitelerde Fen bölümleri, tıp ve mühendislik öğrencileri ilk yıllarında Kimya Laboratuvarı dersi ile karşılaşmaktadır. Kimya Laboratuvarı dersi ile ilgili bu kitapçığın başında öncelikle kimya laboratuvarı için son derece önemli olan kimya laboratuvarında uyulacak kurallar ve laboratuvarında alınması gereken güvenlik önlemleri açıklanmıştır. Laboratuvar güvenliği ile ilgili detaylı bilgiler, verilen web bağlantısındaki kaynakta yer almaktadır. Kimya Laboratuvarında kullanılan malzemelerin tanıtıldığı bölümden sonra, Genel Kimya Ders konuları ile bağlantılı seçilen deneylerin föyleri yer almaktadır.

Laboratuvara ilk geldiğiniz gün, kimya laboratuvarında uyulacak kurallar ve laboratuvarında alınması gereken güvenlik önlemleri konularında bilgilendirilerek, yangın tüpleri, güvenlik duşu, göz yıkama muslukları gibi kaza anında kullanılacak malzeme ve sistemlerin yerleri ve nasıl çalıştıklarını öğreneceksiniz.

Kimya laboratuvarları, her an istenmeyen kazaların yaşanabileceği yerler olması nedeni ile herşeyden önce güvenlik konularındaki bilgilerin öğrenilmesi çok önemlidir. İlk hafta güvenlik kuralları ve önlemleri bölümünü ve her deney haftası deney föyünü mutlaka okuyup öğrenerek geliniz.

Hepinize sağlıklı, başarılı ve kazasız bir eğitim, öğretim dönemi dilerim.

Saygılarımla

Prof. Dr. Serap BEŞLİ

Kimya Bölüm Başkanı

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LABORATORY RULES FOR CHEM 114 GENERAL CHEMISTRY LAB II COURSE

1. The students must attend the General Chemistry Laboratory I course in exact day, time and laboratory which is declared on the course programs's of the department.

2. Attendance compulsion for the experiments is 100%. If any of the experiments are not done, the student is deemed to have failed that course.

3. During the lab course, the students have to wear their lab coat, lab goggles and latex gloves. The students must bring their own lab coat, lab goggles and latex gloves to the lab. If the student does not bring lab coat, lab goggles and latex gloves to the lab or does not use these items in the lab, she/he will not be allowed to attend the lab. The experiment will be marked as failed.

4. If the student is late more than 5 minutes to the lab course, she/he will not to be allowed to attend the lab. and the experiment.

5. During the lab course, it is strictly forbidden to use mobile phone. The students do not leave the lab and experiment set without permission of the lab assistant.

6. In order to provide life safety and to ensure the success of experiment in a safe manner, hand jokes between the students is strictly forbidden. Also, touching to chemicals without gloves, sniffing and tasting them is dangerous and strictly forbidden.

7. In every experiment, chemicals, and materials necessary for the experiment will be supplied to the students by the lab assistants. The students must not borrow the chemicals and materials from the other students. At the end of each experiment, all the glass or metal materials will be cleaned by the students. Also, the experiment set will be tidy and clean.

8. Before coming to lab, the students should know about the procedure of the experiment and theoretical information.

LABORATORY SAFETY RULES AND CAUTIONS

1. Students must wear lab coat and safety glasses in the lab. Students without lab coat or safety glasses will not be allowed in the lab.
2. Contact lenses are not allowed in the lab. Acid and organic chemical vapors could get in between eye and lenses. Thus, lenses can be glued on the eye in the case of an accident and can be difficult to remove.
3. Wide dresses, sandal type shoes shouldn't be worn in the lab and long hair should be tied.
4. Food, drink and chewing gum are not allowed in the lab.
5. Students should know where the fire extinguisher, first aid cabinet and shower are placed in the lab.
6. Learn the emergency exit from the lab in the case of a fire.
7. Use the shower when your dresses or hair catches fire.
8. Do not run and do not make jokes in the chemistry lab.
9. Benches should not be used to sit on or to leave bags or personal things.
10. Smoke and vapor released during chemical reactions should not be smelled directly.
11. It is not allowed for students to work alone in chemistry labs without assistants or instructors.
12. Do not use Bunsen burners next to flammable chemicals (i.e. ethers)
13. Read the labels carefully on the bottle before you use the chemicals.
14. Read the experimental procedure before coming to the lab. Students who come to lab without knowledge about the experiment could create risks for themselves and other students.
15. In the case of any accident (glass cut, acid/base burn, fainting etc.) immediately inform your assistant or instructor.
16. Do not orientate the test tube toward yourself or your friend. Reaction carried out in the test tube might be dangerous.
17. Water should not be added on concentrated acids. Acids should be added to the water slowly and by stirring.
18. It is forbidden to smell and to taste the chemicals and to pull solutions by mouth when using pipet.
19. Chemicals (solid, liquid or solution) must not be dumped into the sink. Waste bottles in the lab should be used. Learn where the waste bottles in the lab are.
20. Use the "broken glass" labeled container in the lab for the broken glass pieces.
21. Matchstick, litmus paper must not be disposed to the sink.
22. Mercury vapor is invisible and toxic. Mercury releasing from a broken thermometer is very dangerous and should be reported to the lab assistant or instructor immediately.
23. Materials like hot test tube, crucible, and beaker must not hold by naked hand. Tube tongs should be used or left to cool on an asbestos wire.
24. Please use the amount of chemicals given in the procedure. Use of excess amounts can make it difficult to control the reactions or cause side reactions.
25. Left behind chemicals should not be returned to the stock bottles; instead, should be discarded into the waste bottles.

26. Always keep your working area and balance clean. Work clean and be tidy in the lab.
27. Don't change the locations of chemicals during the experiment.
28. At the end of the experiment, hand in all the materials you used to the lab assistant/technician as cleaned.
29. Make sure that gas and water taps are closed before leaving the lab.
30. Wash your hands before leaving the lab.
31. Check the following link for more information:

<http://www.gtu.edu.tr/Files/kimyaBolumu/documents/LabGuvenlik.pdf>

The following cases must be notified firstly to the assistant or lecturer.

BURN: expose the burned area to the tap water (5-10 min.), apply first aid.	CUT / INJURY: Wash with water and apply first aid.
FAINTING: Provide fresh air. Lay down and put the head lower than the body.	FIRE: (Notify the assistant immediately) Put the bunsen burner off. Use shower in case of hair and clothes catches fire. Use the fire extinguisher when necessary.
BLEEDING: compress on the wound, keep the wound above the heart level and get medical help.	CHEMICAL SPILL: clean in a manner appropriate to the chemical. Aqueous solutions can be removed with water. Inform your assistant.
ACID BURNS: Use NaHCO_3 solution BASE BURNS: Use Boric acid or Acetic acid solution	EYE CONTAMINATION: The eye is washed immediately with plenty of water for at least 15 minutes (use the eye-wash shower rooms). Get medical help.

Laboratory Safety Symbols

GHS Labels



Oxidizers - Can burn without air, or can intensify fire in combustible materials.



Explosives - May explode if exposed to fire, heat, shock, friction.



Corrosives - May cause skin burns and permanent eye damage.



Gases Under Pressure - Gas released may be very cold. Gas container may explode if heated.



Flammable if exposed to ignition sources, sparks, heat. Some substances may give off flammable gases.



Toxic to aquatic organisms and may cause long lasting effects in the environment.



Toxic material which may cause life threatening effects even in small amounts and with short exposure.



May cause serious and prolonged health effects on short or long term exposure.



Irritant - May cause irritation (redness, rash) or less serious toxicity

LABORATORY EQUIPMENTS



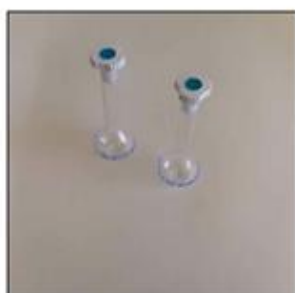
Beakers



Erlenmeyer Flasks



Graduated Cylinders



Volumetric Flasks



Seperatory Funnel



Dropping Funnel



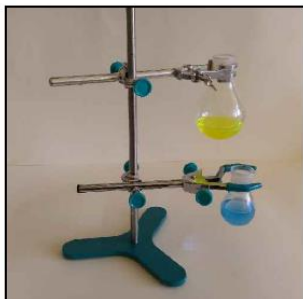
Funnel



Gooch Funnel
(Sintered Glass Filter Funnel)



Suction Flasks



Boiling Flasks



Burette



Bagets (Glass)



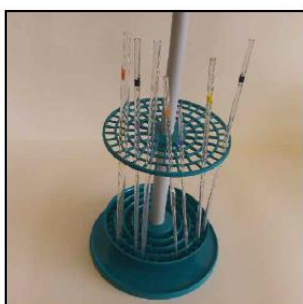
Pipettes



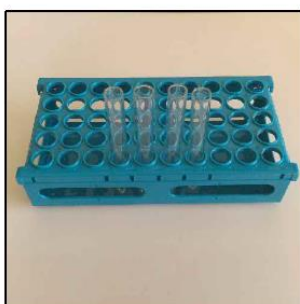
Thermometer



Pi-pump



Pipette Stand



Test Tubes & Stand



Adapters



Distillation Condenser



Stoppers



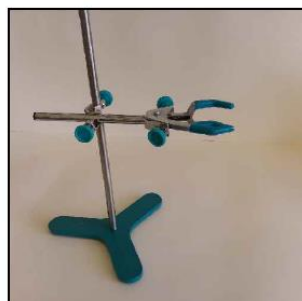
Gas Syringe



Clamp



Bosshead (Clamp Holder)



Laboratory Stand & Clamp



Metal Ring



Forcep



Spoons and Spatulas



Droppers



Baget (Polypropylene)



Magnetic Stir Bar &
Magnetic Stir Bar Retriever



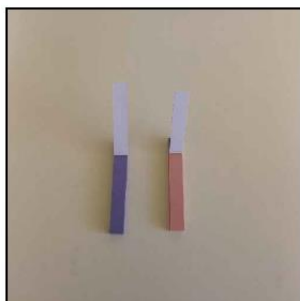
Pipette Bulbs



Magnifier



Clips



Litmus Paper



pH Paper



Filter Paper



Buchner Funnel



Evaporating Dishes



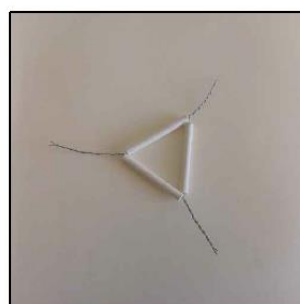
Crucible & Lid



Mortar & Pestle



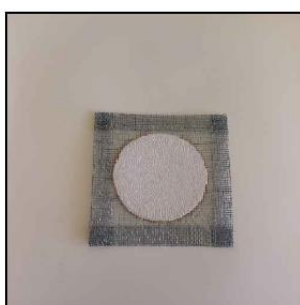
Watch Glass



Ceramic Triangle



Crucible Tong



Wire Gauze



Particulate Respirator



Rubber Tubings



Silicone Tubings



Safety Glasses



Holder for Flask



Stopper Without Hole



Washing Bottles



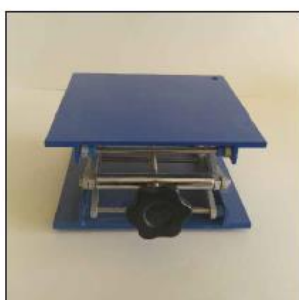
Bunsen Burner



Desiccator



Brushes



Laboratory Jack



Magnetic Stirrer



Heating Mantle

EXPERIMENT 1: LEAD IODIDE PRECIPITATION REACTION

Purpose: Investigation of a precipitation reaction and to learn the general laboratory techniques (solving, crystallization, filtration) used in chemistry laboratories.

Theory: In precipitation reactions, solutions of the anions and cations are mixed and a solid called *precipitate* is obtained as reaction product. Solid reactants and products used in chemical reactions in general may need to be purified. Solving, crystallization, and filtration are the techniques used for purification of solid substances.

Solving: Solving process is made by gradually adding the substance to be solved into the solvent. After each addition, the mixture is stirred with the aid of a stick to dissolve the substance. If there are large pieces of the substance, they are crushed and the mixture is heated if necessary. Glass container do not contact directly to the flame during heating. Asbestos fiber is used for this purpose. When working with organic and flammable solvents, open flame is very dangerous, therefore electrical heaters should be used if possible. If there isn't an electrical heater, an asbestos heating wire should be used necessarily over a low flame very carefully. Excessive boiling should be avoided during heating.

Crystallization: Crystallization is one of the purification methods that is based on the principles of dissolution of a substance in a solvent in which the solubility of the substance is maximum when the solvent is hot and minimum when cold. Therefore, the choice of solvent in crystallization process is very important.

Crystallization Technique: Prepare a hot saturated solution of the crystalline matter in a suitable solvent. This solution is hot filtered into a clean flask with a preheated funnel. The purpose of this filtration is to separate solid particles that are insoluble in hot solvent. The purpose of heating the funnel is to prevent crystallization from saturated hot solution on cold funnel wall. Prior to filtration, funnel must be heated either in an oven or by exposure to a hot steam.

Saturated solution is filtered into the beaker and allowed to cool by covering the beaker with watch glass. The cooling rate is one of the most important factors that affect the size of the crystals. If large crystals are desired to be obtained, cooling should be slow, and small crystals are obtained by fast cooling. Ice bath can be used for rapid cooling.

In some cases, the substance may be separated as an oil without crystallization. In such cases, the solution is heated again to dissolve the oily substance and left to cool very slowly without shaking. To ensure slow cooling, the area around the beaker may be wrapped with a clean towel.

If the crystallization does not occur even though the solution was thoroughly cooled, a glass rod is rubbed against the wall of the beaker for scratching operation, or a seed crystal should be added to the solution. It should also be noted that it is difficult to crystallize from the viscous solution which is concentrated by thoroughly evaporating. So, to concentrate the solution by excessive evaporation should be avoided.

After the formation of crystals they are separated by filtration through a funnel, the crystals are washed with a small amount of a solvent which can not solve crystals in cold and dried.

Filtration: The filtration process is applied in two ways:

a) Simple filtration

For this purpose, the filter paper is folded first in half and then four, after that upper part of filter paper rounded with a scissors and, formed into cone shape by opening one of the plies. Paper is placed on the funnel. Funnel is placed on a ring holder depending on its diameter. After placing a clean beaker under the funnel, the mixture to be filtered is poured in the center of the filter paper with the help of a glass rod. The filtered mixture should never be filled to the higher level of 1 cm below the upper edge of the filter paper.

Pleated paper filter can also be used in the filtering process. Preparation of pleated filter paper is as follows: Round filter paper before folding in half after four (Figure 14.1).



Figure 1.1. Preparation of Pleated Filter Paper

Then the fold is opened, 1 to 2 and 3 to 2 are overlaid and folded to form 4 and 5 corners. Then the sequence (1,5), (3,4), (1,4) and (3,5) are fitted to the corners and folds are created manually pressing the sharp edges. After each folding in the opposite direction of their pleated folds of filter paper is ready to use. (Figure 14.2).

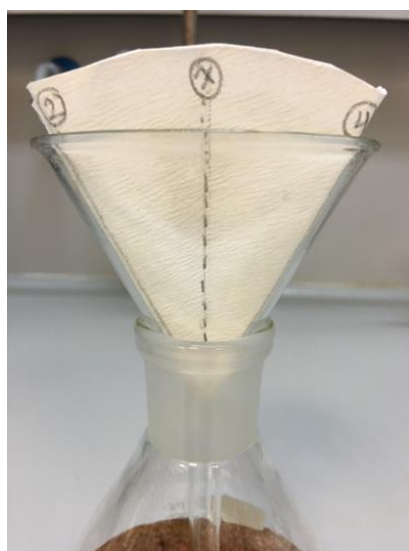


Figure 1.2. Pleated Filter Paper

b) Filtering with Buchner Funnel

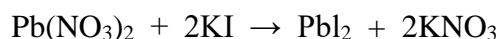
Buchner funnel with filter assembly is shown in Figure 14.3.



Figure 1.3. Filtering with Buchner Funnel

Filter paper which is cut in suitable diameter is placed in a Buchner funnel as shown in Figure 14.3 and vacuum is applied to the system by opening the sink faucet. Meanwhile an amount of distilled water is poured onto the filter paper, so that the paper is firmly adhered to the funnel. Then the mixture be aligned with the cone by means of a glass rod is poured slowly. After the entire mixture transferred into funnel, the crystals on the filter paper are washed and dried with the paper based upon the watch glass.

Reaction Equation:



Materials

Beaker (250 ml) x 2
Glass funnel (big)
Volumetric cylinders (100 ml)
Watch glass
Erlenmeyer (250 ml)
Spatula x 2
Magnetic stirrer with heating
Stirring bar x 2
Filter paper
Glass rod

Chemicals

Lead Nitrate
Potassium Iodide

Procedure:

- 1) Put 1 g of lead nitrate in about 20 ml of water in a beaker
- 2) Dissolve approximately 1 g of potassium iodide in 20 ml water in another beaker and mix these two solutions.
- 3) Add 300 mL of water to the solution until the yellow precipitate dissolved and heat.

Since a large amount of water is necessary, large beaker should be used for the reaction.

- 4) Filter the boiling solution on funnel with a pleated filter paper into another beaker.

Before filtering process the funnel must be heated by holding to vapor.

- 5) Crystallize the collected filtrates by cooling in ice.

- 6) Dry filtered crystals.

Crystals are yellow colored and hexagonal shaped and insoluble in alcohol. The solubility in water is 0.063 g / 100 ml at 20 °C.



Fig. 1.4. Yellow precipitate, PbI_2

Experimental Data:

The mass of $\text{Pb}(\text{NO}_3)_2$ _____

The mass of KI _____

The mass of filtering paper _____

Filter paper + product mass _____

Product mass _____

Calculations: *Show the calculations step by step in lab reports.*

Number of moles of $\text{Pb}(\text{NO}_3)_2$

Number of moles of KI

Name and number of moles of excess reactant

Number of moles of the product (obtained)

Number of moles of the product (theoretical)

% Yield

Questions: *Give answers to the following questions in pre-lab reports.*

- 1) If 25 ml of 0.5 M $\text{Pb}(\text{NO}_3)_2$ solution is added into 50 ml 0.5 M KI solution, how many grams of PbI_2 precipitates ?

EXPERIMENT 2: MOLECULAR WEIGHT DETERMINATION USING FREEZING POINT DEPRESSION

Purpose: Freezing point determination and molecular weight determination of a solute from the freezing point depression of solvent. Determination of molecular weight of sulfur from the freezing point depression of naphthalene.

Theory: When a pure solid is heated, temperature of the solid increases until melting point of the solid is reached and temperature stays constant during fusion. Temperature starts to increase after the phase change (from solid to liquid) is complete.

Temperature increases of solid and liquid states are different, since heat capacities of solid and liquid states are not same. As it can be seen in temperature-time graph (Figure 12.1) below liquid starts to turn into solid at temperature T_1 and the temperature remains constant during this turning period (time $t_1 - t_2$).

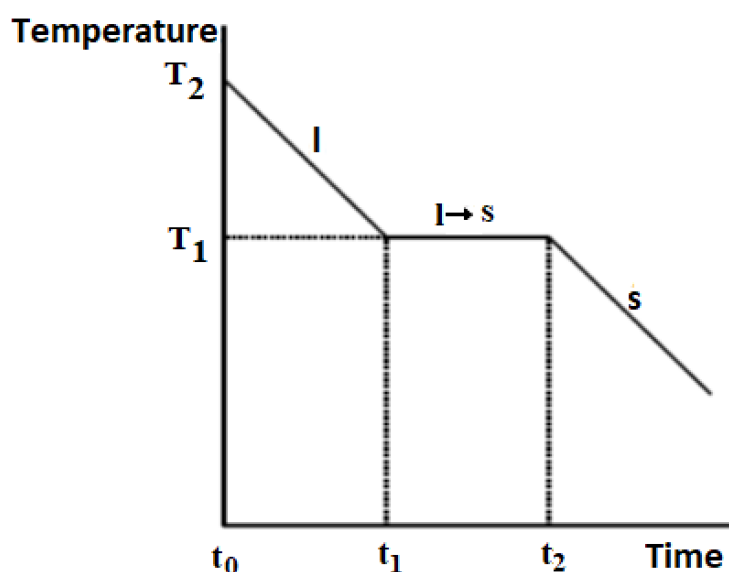


Fig. 2.1. Temperature - time graph (cooling curve).

During freezing, as the particles pass from liquid to solid, the potential energy of the material starts to decrease. For this reason, the formation of solid particles during freezing forms heat energy, and this energy that meets the heat energy dissipated by cooling. As a result, the temperature remains constant until the freezing is complete. This helps us to determine the freezing point of a substance. The melting point of a substance is the temperature at which the liquid is equilibrated together. For example, at 1 atm pressure, ice melts at 0°C . During melting period solids become irregular and transfer into liquid from a regular and certain crystal structure. The amount of heat required to melt one mole of a solid is called molar heat of fusion.

Then, the heat given will increase the average kinetic energy of the liquid molecules and the temperature of the liquid will increase until the temperature reaches the boiling point. The

temperature at which the vapor pressure of a liquid is equal to the atmospheric pressure is called boiling point. At this temperature the liquid starts to boil. The boiling liquid evaporates and over time the whole liquid becomes vapor. The subsequent heat is used to increase the kinetic energy of the vapor molecules. The amount of heat required to evaporate one mole of liquid is called the molar heat of vaporization. The temperature, called as condensation temperature, at which the gas is converted into liquid is the same as the temperature at which the liquid is converted into the liquid (boiling point). In the similar case, the temperature (freezing point) at which the liquid turns into solid is the same as the temperature at which the liquid turns into liquid (melting point). It is known that, compared to pure solvents, the total vapor pressure of unvolatile solutions of these solvents is lower. The decrease in vapor pressure in such solutions, causes the boiling point to rise and the freezing point to decrease. Such properties of solutions are called "colligative properties".

Colligative properties are numerical properties that are not depend on the structure and chemical properties of the material but only depend on the structure of the molecule. These properties include lowering of vapor pressure, depression of freezing point, boiling point elevation and osmotic press. The colligative properties vary depending on the solute / solvent particle ratio in the solution. In general, the addition of a solute raises the boiling point of the solvent while reducing the freezing point. The descent of the freezing point for a given solvent is directly proportional to the concentration of the solute present in the solvent.

$$\Delta T = K_d \times m$$

$$\Delta T = \text{Freezing point depression}$$

$$K_d = \text{Freezing point depression constant of solvent}$$

$$m = \text{molality}$$

Molality is the number of moles of solute in 1000 g of solvent.

$$\text{Molality (m)} = \text{number moles of solute, } n / 1000 \text{ g solvent}$$

Measurements related to freezing point depression as well as boiling point elevation can be used to determine the molecular weights of substances. For instance, a certain amount of substance with an unknown molecular weight is dissolved in a solvent with a known freezing point depression constant. This solution is allowed to freeze and the freezing point is determined. Freezing point depression and molality of the solution are determined.

In this experiment naphthalene is solvent and sulfur is solute. If one mole of sulfur is dissolved in 1000 grams of naphthalene, freezing point of the solvent decreases by 6.9°C ($K_d = 6.9^\circ\text{C/molal}$). A known amount of sulfur will be dissolved in a known amount of naphthalene and molecular weight of sulfur will be determined.

Materials

Beaker (400 mL)
Two holed cork
Test tube
Magnetic stirrer and stirring bar
100 °C (0.1 °C scaled) thermometer
Clamp, clamp holder and stand

Chemicals

Naphthalene
Sulfur

Procedure:

- 1) Place the thermometer in a two holed cork in a way that 70°C and above of thermometer scala can be read.
- 2) Make sure that the tip of the thermometer does not touch bottom (otherwise it would be broken).
- 3) Weigh 5 g of naphthalene and record the amount of solvent, **m₂**.
- 4) Pour all naphthalene in the bottom of a big test tube. Make sure that the test tube is clean and dry before filling.
- 5) Cover the tube with the two holed cork with thermometer and a wire stirrer placed.
- 6) Place the tube in a beaker containing water.
- 7) Heat the beaker slowly until naphthalene completely melts. Melting point is observed around 80°C.
- 8) Take the bunsen burner under the beaker and put it off. Record the temperature at every 30 second while stirring continuously. Continue this procedure till the temperature drops to 75°C.
- 9) Weigh 1 g of sulfur (record the amount of solute, **m_L**) and add on the cooled solid naphthalene.
- 10) Place the thermometer and stirrer, and heat in a water bath until naphthalene and sulfur melt completely.
- 11) After turning the bunsen burner off and record the temperature drop at every 30 second in the 85-70°C range.
- 12) Do your calculations using your experimental data.

To clean the test tube, heat in water bath once more until all contents melt. Take the thermometer and stirrer out after contents of the test tube completely melted. Empty the melted naphthalene in the naphthalene labeled beaker. Never pour the melted naphthalene in the sink.

Pre-lab Questions: Give answers to the following questions in pre-lab reports.

- 1) Define the following terms briefly:
 - Colligative properties
 - Molar heat of evaporation

Results and Discussion: Show the calculations step by step in lab reports and draw the necessary curves.

- 1) Draw the cooling curves for pure naphthalene and sulphur-naphthalene solution on a graph paper.

Naphthalene (solvent) weight (**m₂**)

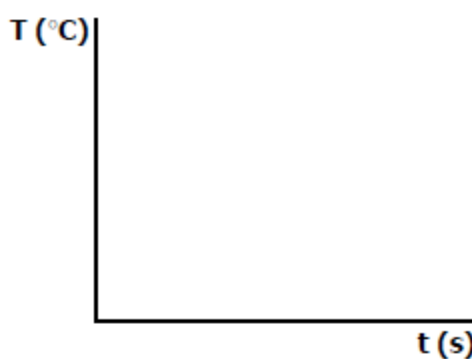
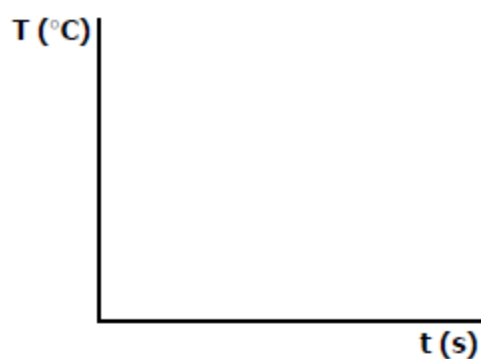
Cooling curve data of pure Naphthalene

t (time, s)	T (temperature, °C)	t (time, s)	T (temperature, °C)
.....
.....
.....
.....
.....
.....
.....
.....
.....

Sulfur (solute) weight(**m₁**)

Cooling curve data of Sulphur solution in Naphthalene

t (time, s)	T (temperature, °C)	t (time, s)	T (temperature, °C)
.....
.....
.....
.....
.....
.....
.....
.....
.....



2) Determine the freezing points from the cooling curves.

Freezing point of pure naphthalene (T_1)

.....

Freezing point of the solution (T_2)

.....

3) K_d (Naphthalene) is 6.9°C/molal . Calculate the molality of the solution.

Freezing point depression (ΔT)

.....

Molality of the solution (m)

.....

4) Calculate the molecular weight of sulfur using Molality (m), m_1 ve m_2 data.

Molecular weight of sulfur (MW)

.....

5) Atomic mass of sulfur is 32 g/mol . write the molecular formula of sulfur as S_n using the molecular weight value you found.

S_n

6) Look at the correct molecular formula of sulfur in your book. Calculate the theoretical molecular weight of sulphur and percentage error of your experiment.

% Error in MW

Report Questions: Give answers to the following questions in lab reports.

1. What is the percentage error in the freezing point value of naphthalene?
2. What is the advantage of using experimental freezing point value for naphthalene to determine the molecular weight of sulfur?

EXPERIMENT 3: A KINETIC STUDY OF IODINE CLOCK REACTION

Purpose: To determine the effect of concentration of reactants on the reaction rates.

Theory: The rate equation for a chemical reaction $A + B \rightarrow \text{products}$ may be expressed as

$$R = k [A]^m [B]^n$$

R represents the rate of reaction in terms of the change in concentrations of reactants or products ($\Delta \text{Concentration}$) divided by the time interval (Δtime)

$$R = - [\Delta A] / \Delta t = - [\Delta B] / \Delta t = [\Delta \text{product}] / \Delta t$$

k is known as the specific rate constant, which is specific to the reaction. The rate constant is dependent on the temperature of the solution.

[A] and [B] gives the molar concentrations of the reactants.

m+n define the order of the reaction.

As the reaction proceeds, the rate varies; therefore **R** indicates the initial rate of reaction. The order of any reaction should be determined experimentally, and can not be deduced from the balanced form of the reaction.

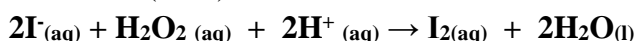
The rate of any reaction depends on several parameters, some of them are:

- Concentration of the reactants
- Temperature
- Presence of a catalyst

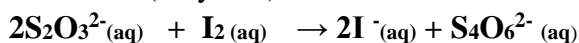
In this experiment, the effects of concentration on the rate of the following reactions will be investigated.

Multiple reactions

Reaction 1 (slow):



Reaction 2 (very fast):



Reaction 3:



The iodine (I_2), produced in reaction (1) is absorbed immediately by thiosulfate ($\text{S}_2\text{O}_3^{2-}$) ion in the solution (reaction 2). When $\text{S}_2\text{O}_3^{2-}$ used up completely the concentration of I_2 is increased, which can be observed easily by the formation of deep blue color (reaction 3).

The first reaction is rate determining step. The rate of the reaction is dependent on the concentrations of the iodide ion (I^-) and hydrogen peroxide (H_2O_2). The spectator ions are left off the reaction. Hence, the **rate law** (concentration dependence) for the reaction can be written as:

$$R = k [\text{I}^-]^x [\text{H}_2\text{O}_2]^y$$

The aim is to find out x and y for the reaction and the order of the reaction.

Materials

Beakers (100 mL) x 6
 Volumetric flask (50 mLx1) and (25 mLx3)
 Graduated cylinders (50 mLx1) and (10 mLx1)
 Graduated pipette (5 mLx3) and (2 mLx1)
 Pipette pump (green)
 Glass rodx3
 Spatula

Chemicals

0.2 M KI
 3% H₂O₂
 1 M H₂SO₄
 0.1 M Na₂S₂O₃
 Starch
 Distilled water

Precautions:

H₂O₂: Solutions are irritant to mucous membranes, eyes and skin. If swallowed, evolution of oxygen may cause nausea, vomiting and internal bleeding. Avoid contact with eyes and skin.

Procedure

Three different trials will be carried out. In each trial, the concentration of only one reagent will be changed. The required amount of water will be added to keep the total volume same ($V_T=50$ mL) in each trial.

Trial	Mixture A				Mixture B	
	KI (0.2 M), mL	Na ₂ S ₂ O ₃ (0.1 M), mL	Distilled water, mL	Starch (spatula)	H ₂ O ₂ (3%), mL	H ₂ SO ₄ (1 M), mL
1	10	3	31,5	1	2,5	3
2	5	3	36,5	1	2,5	3
3	5	3	34	1	5	3

1) In three beakers first combine indicated volumes of KI, Na₂S₂O₃, distilled water and one spatula starch indicator as seen in the table above (Mixture A).

2) In the other three beakers combine the indicated volumes of H₂O₂ and H₂SO₄ as seen in the table above (Mixture B)

3) Add mixture B to mixture A and immediately start the stopwatch and wait for the color change to appear.

4) When the color change appears, stop the stopwatch and note the time it takes for the color change to appear.

The dependence of the reaction rate on the concentration of the reactants can be observed by comparing the results.

Trail	$[I^-]_0$, (M)	$[H_2O_2]_0$, (M)	Reaction time (s), Δt	Reaction rate (Ms^{-1}) $= [\Delta I_2] / \Delta t$
1				
2				
3				

* $[I^-]_0$: Initial concentration of iodine ions.

* $[H_2O_2]_0$: Initial concentration of hydrogen peroxide.

Pre-lab Questions: Give answers to the following questions in pre-lab reports.

- 1) Which factors affect the reaction rate? Explain how each factor affect the reaction rate.
- 2) Calculate the molar concentration of 3% H_2O_2 solution.
- 3) Calculate the initial concentration of iodine ion and hydrogen peroxide and write them in the table above.

Report Questions: Give answers to the following questions in lab reports.

- 1a) Using the data obtained from the experiment, determine the rate law expression for the reaction.
- 1b) What is the overall order of the reaction?
- 1c) Find the rate constant, k ?
- 2a) Using the data given below, determine the rate law expression for the reaction:



[A]	[B]	Reaction rate (Ms^{-1})
0.1 M	0.1 M	2.5
0.2 M	0.1 M	5
0.2 M	0.3 M	45

- 2b) What is the overall order of the reaction?
- 2c) Find the rate constant, k ?
- 2d) What is the rate of the reaction when $[A]=0.12$ M and $[B]=0.06$ M.

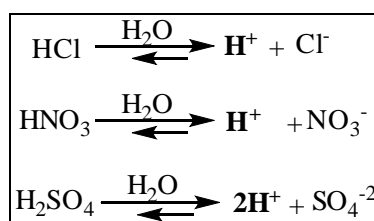
EXPERIMENT 4: ACID - BASE TITRATION

Purpose: Preparation of acid and base solutions, learning of neutralization reaction.

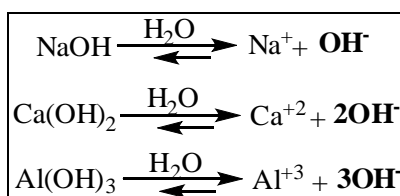
Theory: We encounter the concept of acids and bases in our daily life. For example, acid rain phenomenon is known as a current environmental issue. Acids and bases are one of the most important topics in chemistry since a majority of the chemical reaction is the acid and base reaction.

Despite the wide variety of definitions of acids and bases were made, Arrhenius, Bronsted-Lowry and Lewis definitions are commonly used today.

Acid: Acid turns blue litmus paper red; is active with some metals (active metals) exposing hydrogen gas; has sour taste and forms hydrogen ions in the aqueous solution.

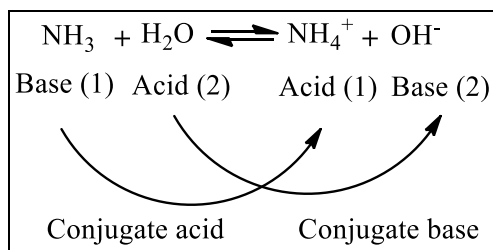
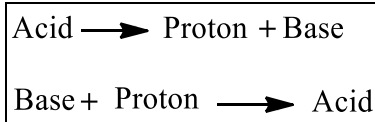


Base: Base turns red litmus paper blue and neutralizes acid. It forms OH^- ions in aqueous solution; Bases form water and carbonated compounds reacting with CO_2 . They don't react with many of metal (except amphoteric metals).



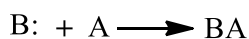
Lowry-Bronsted Acid-Base Definition:

Acids are substances which give proton, bases are substances which take proton. According to scientists conjugate base is formed when acid gives proton and similarly conjugate acid is formed when base takes proton.



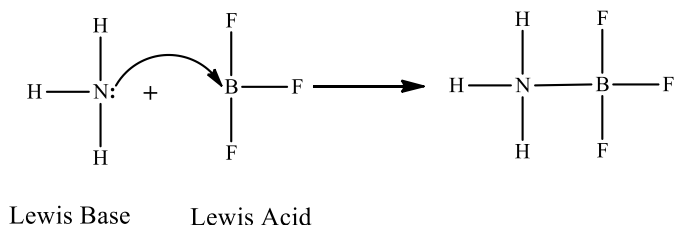
Lewis Acid-Base Definition:

Acid is a substance which takes an electron or accepts electron pair. Base is a substance which gives electron or carries an electron pair.



A = Lewis Acid

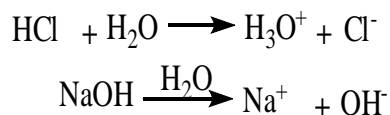
B: = Lewis Base



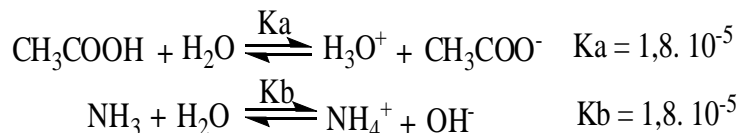
If acid gives only one proton, it is named as monoprotic, mono functional, mono basic, mono equivalent. According to ionization capability in their aqueous solutions, acids and bases are classified as;

- a. Strong
- b. Mild
- c. Very mild

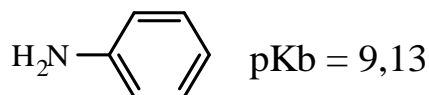
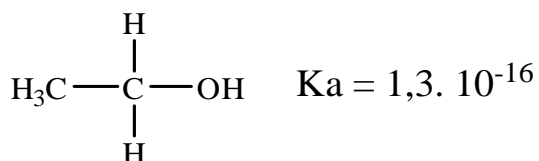
a) Strong Acids and Bases: Acids or bases which are 100% ionizable in aqueous solution is called "strong acid or base". It is accepted that H_3O^+ ions completely originated from acid in strong acid solutions and OH^- ions completely originated from bases in strong base solutions.



b) Weak Acids and Bases (1): Weak acids or bases cannot be ionized 100% in their aqueous solutions. Ionization of acids and bases is a chemical equilibrium reaction either with acidity constant, K_a or basicity constant, K_b .



c) Weak Acids and Bases (2): Weak acids or bases can be partially ionized in their aqueous solution. Their K_a and K_b constants are very low whereas their pK_a and pK_b values are very high.



Volumetric Analysis:

Analysis of the synthesized or natural samples is one of the fundamental subjects of the chemistry. In chemical analysis, identification of the analytes in a sample called “analysis” and determination the amounts of the analytes in a sample called “analysis”. There are two basic methods for quantitative analysis of the chemical samples in which no instruments are used. These methods are “gravimetric analysis” and “volumetric analysis”. In gravimetric analysis, amount of a known (identified) substance is determined by measurement of mass. For this, a water insoluble compound of the substance is prepared via a chemical reaction. And mass of the compound is used to calculate the amount of this substance. Volumetric analysis is based on the measurement of the volume of a solution prepared in exact concentration of a substance which gives a complete reaction with the analyte. By using the measured volume, the concentration or mass of the substance can be calculated.

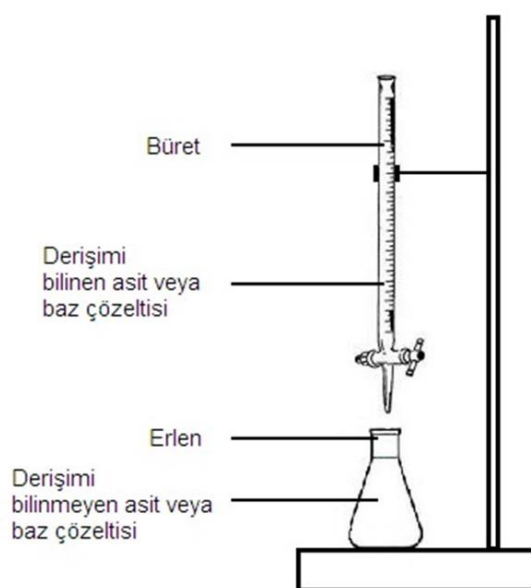


Figure 4.1. Titration setup

Titration, also known as titrimetry, is a common laboratory method of quantitative chemical analysis that is used to determine the unknown concentration of an identified analyte. Since volume measurements play a key role in titration, it is also known as volumetric analysis.

In titrations, known concentration and volume of titrant reacts with a solution of analyte to determine the concentration. Standard solutions are used as titrant to determine the unknown concentrations of substances. **Standard solution** is a solution containing a precisely known concentration of an element or a substance. Chemical substances generally contain impurities, therefore solution concentration can not be known precisely. Titrant solutions are standardized by titration of primary standard (pure, high molecular weight,..) substances.

Volume of titrant used to reach the equivalent point is called titration volume.

The equivalence point, or stoichiometric point, of a chemical reaction is the point at which chemically equivalent quantities of acid and base have been mixed. In other words, the moles of acid are equivalent to the moles of base. It can be found by means of an indicator, most often phenolphthalein. In a reaction, the equivalence of the reactants as well as products is conserved. The endpoint refers to the point at which the indicator changes colour in a colourimetric titration. Indicators are substances which show whether the substance tested is

basic or acidic. pH indicator is a chemical detector for protons in acid-base titrations. The reagent of known concentration and volume used in titrations.

An **acid–base reaction** is a chemical reaction that occurs between an acid and a base. It is shown as;



Reaction is balanced. If the reacting acid and base are both too strong, pH would be 7 at the time that the titration was completed.

· **Equivalence factor:** The number of hydrogen and hydroxide ions that can be substituted for the acids and bases (e).

· **Equivalent weight:** The ratio of the molecular weight to equivalence factor (E).

· **Amount of equivalent gram:** The ratio of the grams of material to equivalent weight (E_A).

· **Normality:** Normality is a measure of concentration equal to the gram equivalent weight per liter of solution (N).

Volume of solution: liter (V).

Amount of dissolved matter: gram (m).

Molecular weight: g/mol (M_A)

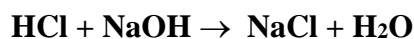
These terms using;

$$N = \frac{E_A}{V} = \frac{m/E}{V} = \frac{m}{M_A/e \cdot V}$$

According to principle of equivalence; $N_a \cdot V_a = N_b \cdot V_b$

Students will find the amount of acid to neutralize 25 mL of 1 M sodium hydroxide. Acid and base will react to form sodium chloride which can be crystallized further.

Neutralization reaction;



Materials	Chemicals
Erlenmeyer flask (250 mL) x 5	HCl solution
Beaker (100 ml)	1 M NaOH solution
Burette (25 ml or 50 mL)	Methyl orange (indicator)
Volumetric cylinder (100 mL)	
Spatula	
Dropper	
Clamp, clamp holder and stand	

Experimental Procedure:

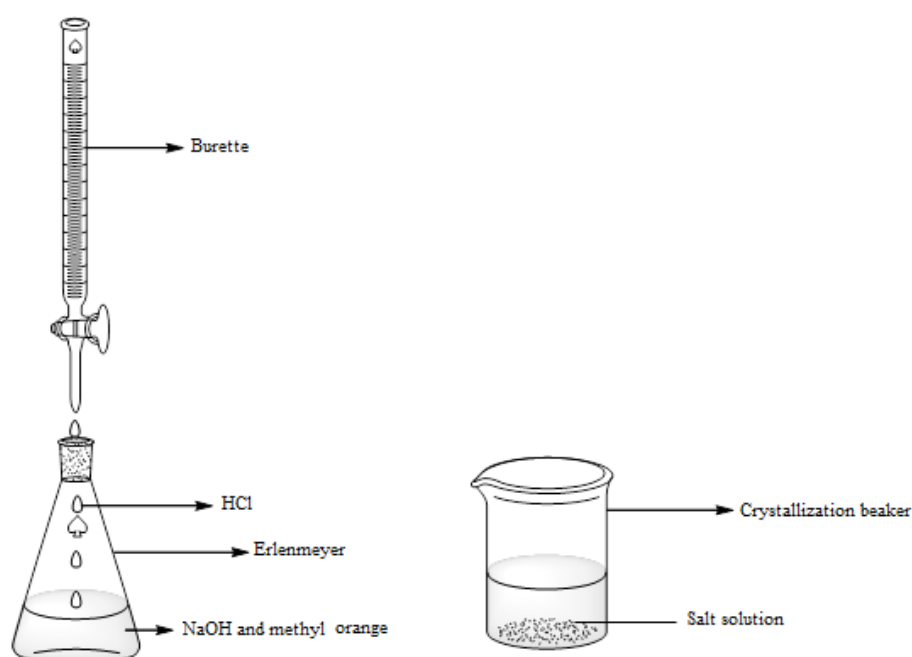


Figure 4.2. Experimental set up for pH titration

In this experiment, sodium hydroxide forms sodium chloride-soluble salt with hydrochloric acid neutralization. This solution is concentrated and crystallized in the crystallization vessel.

1. Prepare 25 ml 1 M NaOH solution in an erlenmeyer.
2. Add two drops of methyl orange to the NaOH solution and mix them.
3. Full the burette with unknown molarity HCl solution end of the zero point.

4. Add in a small volume of hydrochloric acid solution onto sodium hydroxide solution. Shake after each addition. Continue the addition of acid until a red solution appears.
5. Please read through the burette volume of added acid.
6. Repeat titration (4. step) two more times. And use the average value of these 3 titrations for your calculations.

Cautions:

Wear protective gloves, gowns, glasses and a mask.

Avoid breathing vapors.

Results and discussion:

HCl quantities which is needed neutralization.

	1.Titration	2.Titration	3.Titration
Used NaOH volume (mL)			
Used HCl volume (mL)			
Normality of the HCl solution			
Normality of the NaOH solution			
Milliequivalent gram of used HCl			
Milliequivalent gram of used NaOH			

Questions:

1. Explain acids and bases definitions of Arrhenius, Bronsted-Lowry and Lewis.
2. Explain definitions of Titration, The equivalence point, Indicator, titrant briefly?
3. A 25 ml solution of 0.5 M NaOH is titrated until neutralized into a 50 ml sample of HCl.
What was the concentration of the HCl?

EXPERIMENT 5: DETERMINATION OF REACTION ENTHALPY

Purpose: To investigate heat changes during chemical and physical events.

Theory: Thermochemistry is concerned with chemical phenomena accompanying changes in heat. The amount of heat exchanged with the surroundings is an important property of a chemical reaction. The amount of heat absorbed or released during a reaction under constant pressure is called "enthalpy" and is represented by the variable H . When the pressure of the system doesn't change, the change in enthalpy of a system equals the heat exchanged with the surroundings: $H=q$

If a reaction gives heat to its environment, that reaction is exothermic and enthalpy change (ΔH) is negative. If a reaction receives heat from its environment, that reaction is endothermic and enthalpy change (ΔH) is positive. Reaction enthalpy can be classified into more specific categories:

Formation enthalpy is the enthalpy change during the formation of 1 mole substance from its elements in standard conditions.

Enthalpy of combustion is the enthalpy change during combustion of a 1 mole substance in the presence of oxygen.

Enthalpy of solution, vaporization, melting and sublimation is related with changes of state of matter or related with dissociations of molecules and ions.

Enthalpy of neutralization is the heat released during acid-base reactions when one mole of water is produced.

Heat measurements are performed by carrying out a chemical reaction in a calorimeter (Figure 1). The reactants are stirred in a container made of polyurethane foam and the temperature change is measured. Polyurethane foam is a good thermal insulator that the cup and its content are considered in isolation.

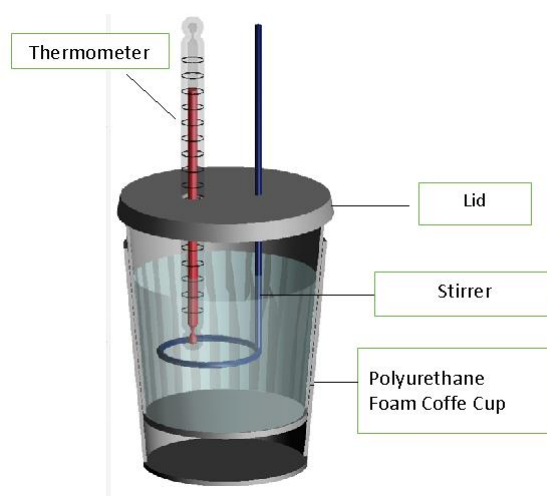


Figure 5.1. Representation of coffe cup calorimeter.

The amount of heat lost or gained in chemical reactions can be calculated by the following formula:

$$q = m \cdot c \cdot \Delta T$$

where **m** is the mass of solution (g), **c** is the specific heat (cal/g °C, J/g °C), and **ΔT** is the temperature change of the solution ($T_{\text{final}} - T_{\text{initial}}$).

The heat gained by calorimeter is calculated by following formula:

$$q_{\text{cal}} = \text{heat capacity of calorimeter} \cdot \Delta T$$

The heat capacity of a calorimeter is given in cal / °C or J / °C. In this experiment, first the heat capacity of a calorimeter will be predicted, and then the heat of fusion of ice and heat of neutralization will be measured.

Materials

Beaker (100 ml) x2
Volumetric cylinder (100 ml) x 2
Calorimeter
Thermometer
2 piece Styrofoam coffee cup

Chemicals

30 ml 2 M HCl solution
30 ml 2 M NaOH solution
1 piece of ice cube
Distilled water

Precautions:

- If an acid is to be diluted, pour acid slowly into the water with constant stirring. Never add water to acid.
- Never inhale gaseous fumes or position your nose directly above the sample. If you need to determine the odor of a gas, gently fan a small amount of the vapor toward your nose with your hand.

Procedure:**A) Determining the heat capacity of a calorimeter**

- 1) Put 50 ml water into the calorimeter and measure the temperature 3 times at 30 second intervals.
- 2) Put 60-70 ml water into a beaker and heat it about 50°C.
- 3) Carefully move the hot water from the heater and measure the temperature 3 times at 30 second intervals.
- 4) As soon as measuring the hot water's temperature, add the hot water into the calorimeter containing cold water. After mixing, measure the temperature 3 times at 30 second intervals.
- 5) Calculate the heat capacity of the calorimeter.

B) Determining the heat of fusion of ice

- 1) Put 60-70 ml water into a beaker and heat it to around 50°C.
- 2) Carefully move the hot water from the heater and add it into the calorimeter and measure the temperature 3 or 4 times at 30 second intervals.

- 3) Put an ice cube which its temperature is already known (ask your assistant), to the hot water in the calorimeter. Stir and then measure the final temperature after the ice is totally melt.
- 4) Precisely measure the final volume of water.
- 5) Calculate the heat required to melt the ice cube (heat of fusion).

C) Determining the heat of neutralization

- 1) Place 30 ml of 2 M HCl solution into the calorimeter and measure the temperature 3 or 4 times at 30 second intervals (thermometer should be dry and clean).
- 2) Place 30 ml of 2 M NaOH solution into the calorimeter and measure the temperature 3 or 4 times at 30 second intervals (thermometer should be dry and clean).
- 3) Add NaOH solution into HCl solution in the calorimeter, stir and measure the temperature 4 times at 30 second intervals.
- 4) Calculate the heat of neutralization for 1 mole of water produced.

The solution obtained after neutralization has a density of 1.02 g/ml and its specific heat is 0.95 cal/g°C.

Pre-lab Questions: Give answers to the following questions in pre-lab reports.

- 1) Define the following terms in your own words
 - Enthalpy
 - Exothermic reaction
 - Endothermic reaction
 - Heat capacity
- 2) Determine the final temperature when 100 g of ice at -9°C is mixed with 600 g of water at 30°C .
 ($c_{\text{ice}} = 0.5 \text{ cal/g}^{\circ}\text{C}$, $c_{\text{water}} = 1 \text{ cal/g}^{\circ}\text{C}$, $\Delta H = 80 \text{ cal/g}$)

Report: Show the calculations step by step in lab reports.

A) Determining the heat capacity of a calorimeter

Temperature of Cold Water

Temperature of Hot Water

Temperature of Mixture

Heat lost by hot water	_____	cal
Heat gained by cold water	_____	cal
Heat gained by calorimeter	_____	cal
Heat capacity of calorimeter	_____	cal/°C

B) Determining the heat of fusion of ice

Volume of hot water	_____	Mass of ice	_____
Temperature of hot water	_____	Initial temperature of ice	_____
Temperature after mixing	_____	Temperature after mixing	_____
Temperature change	_____	Temperature change	_____
Heat lost by water	_____	Heat gained by ice	_____
Heat lost by calorimeter	_____		

Heat for melting ice	_____	cal
Heat of fusion	_____	cal/g
Molar heat of fusion	_____	cal/mole

C) Determining the heat of neutralization

Temperature of NaOH solution	_____	Temperature of HCl solution	_____
_____		_____	
_____		_____	

Temperature of Mixture

Heat gained by solution	_____	cal
Heat gained by calorimeter	_____	cal
Total heat released	_____	cal
Mole number of water	_____	mole
Heat of neutralization	_____	cal/mole